1.0 ETHOS 900

1.1 INTRODUCTION

In the world of microwave sample preparation, Milestone continue to quietly lead the evolution of technology.
Our only focus is to provide you with the most technologically advanced instrumentation for total microwave sample preparation.
In 1994, the ETHOS project was started to develop a new generation of microwave chemistry systems based on the experience of several thousands users worldwide, who process some of the most difficult samples (ores, coal, fly ash, ceramics, and plastics).
Milestone R&D engineers were given the goal to develop the safest, highest performing and most flexible microwave platform for laboratory applications.
No limits were set, only goals.
This is ethical.
This is ETHOS.

1.2 TECHNICAL SPECIFICATIONS

- Microwave labstation for quality control for digestion, drying and evaporation/concentration procedures.
- Patented self-sealing explosion resistant door.
- Single magnetron with static microwave diffuser for homogeneous microwave distribution.
- Installed microwave power 900Watt.
- Delivered microwave power 900Watt in 10Watt increments.
- Variable microwave pulse width from 0,1” to 5”.
- Unpulsed microwave emission at 250Watt.
- Microwave cavity all made of 18/8 stainless steel housing with multilayer Teflon plasma coating applied at 350°C.
- Dimensions 35cm x 35cm x 35cm (ca. 45L volume).
- Built-in exhaust system with automatic constant flow control, acid and organic solvent resistant; with flow rate 5m^3/minute.
- Control terminal “240”, with high resolution graphic (128 x 240) LCD display black/white with backlight and flat keyboard; alphanumeric display, 12 special function keys.
- Storage of up to 20 programs; each program has up to 10 time/power/pressure/internal temperature/external temperature steps.
- Outputs: 1 parallel Centronix for external printer (to print out reports); 2 serial ports (RS-232) for connecting PC (for data base reporting and programming of the unit), balance (for weighing data storage); 1 additional port for analog data (for connecting a laboratory recorder).
- Dimensions of the microwave unit: 55cm large x 55cm long and 70cm high.
- Power supply 230V, 50 or 60Hz, 7A, 1.600Watt.
- Weight approximately 85kg.

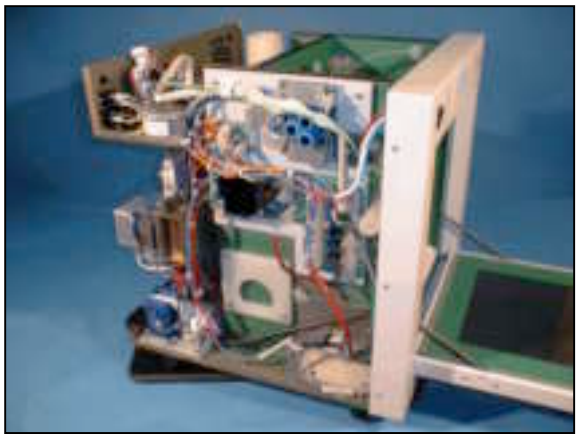
P/N	Description	Power supply
44006	ETHOS 900 complete	220V/50Hz
44056	ETHOS 900 complete	220V/60Hz

1.2.1 A LOOK AT THE HIDDEN SIDE OF THE ETHOS UNIT

The most important safety features are the ones you don't see from the outside.
For this reasons Milestone lets you see under the cabinet cover of the ETHOS.

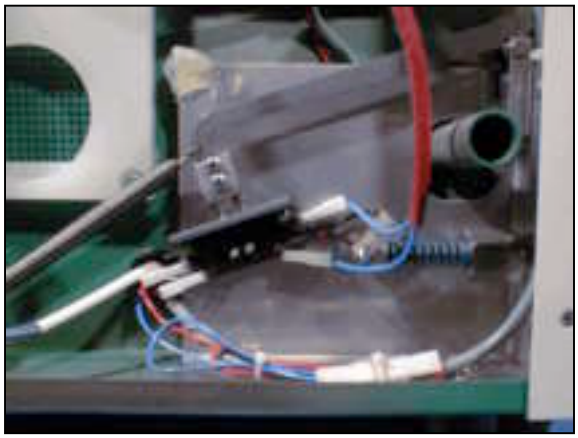
1.2.2 SAFETY THROUGH MECHANICAL DESIGN

The ETHOS is not a kitchen type nor a commercially modified kitchen microwave unit; it is a rugged, heavy duty professional system designed to guarantee years and years of trouble free and safe operation.
The hardware The microwave cavity is quite large, to accommodate different types of accessories.
Dimensions are 35cm x 35cm x 35cm, for a volume of approximately 43L.
The door is, as standard feature, made of stainless steel; however, on request (ETHOS 1600 only), it is possible to have a door with a high impact shock resistant 27cm x 27cm glass window for easy viewing of the illuminated cavity (picture).
is manufactured with 18/8 stainless steel, 1,5mm gauge (picture).



1.2.3 SAFETY THROUGH PATENTED DOOR DESIGN

The door design is the most critical issue when developing new microwave hardware.
Mechanical strength and perfect sealing of the cavity to avoid microwave leaks are prerequisites.
Milestone's engineers, on the basis of the "movable door" principle used in high pressure autoclaves, designed a door with an innovative opening/self resealing mechanism.
The door frame is mounted on four spring-loaded high strength steel bars (picture).

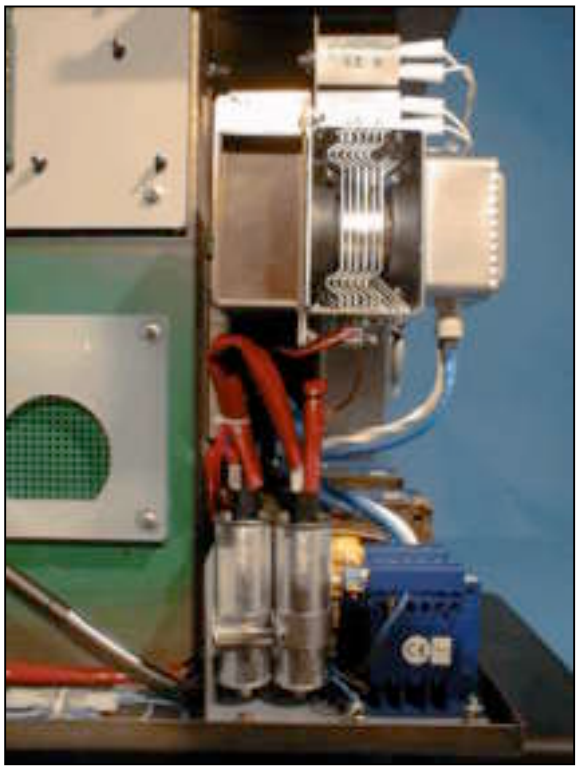


In the event of a sudden overpressure inside the cavity, the entire door opens horizontally ("moves out") presenting a large surface area for immediate and safe release of the overpressure.
Microwave power is instantaneously cut off.
Immediately afterward, the door is "pulled back" by the springs, resealing the cavity.
The microwave cavity door opens downwards to a flat horizontal position, forming a stable work platform for handling microwave accessories before and after digestion procedures.

1.2.4 SAFETY TROUGH ELECTRICAL DESIGN

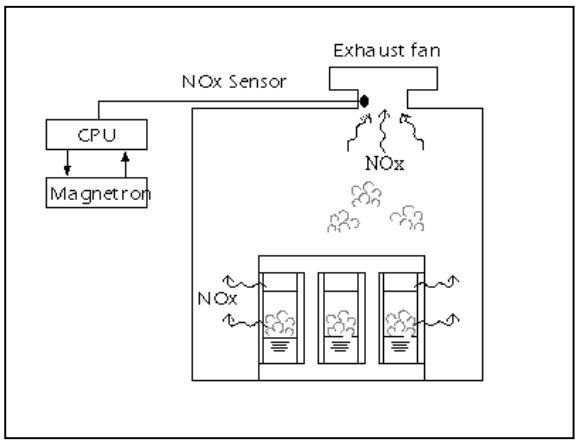
The ETHOS microwave platform is equipped with a total of 10 microswitches to assure the highest safety conditions.
Four (two on each side of the door) fast release microswitches instantaneously interrupt the microwave emission whenever the door is opened (previous picture).
One safety switch on the cabinet cuts off microwave emission whenever the cabinet is detached (picture).

Two thermal switches on magnetron's body and other two on both wave guides cut off power in case of overheating (picture).



1.2.5 SAFETY THROUGH NO_X MONITORING

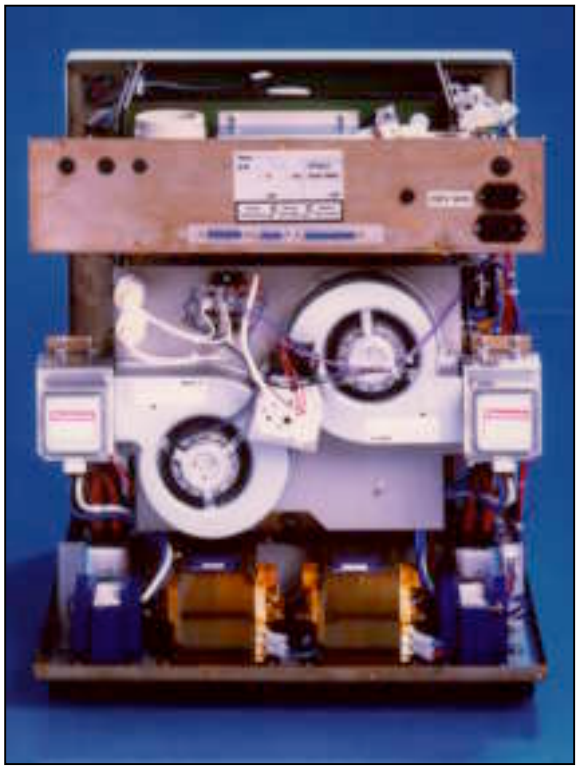
When HNO_3 is used in the digestion process NO_X gases are generated.
NO_X gases may diffuse through the vessel walls or they are released in case of vessel overpressurization, in the microwave cavity (picture).



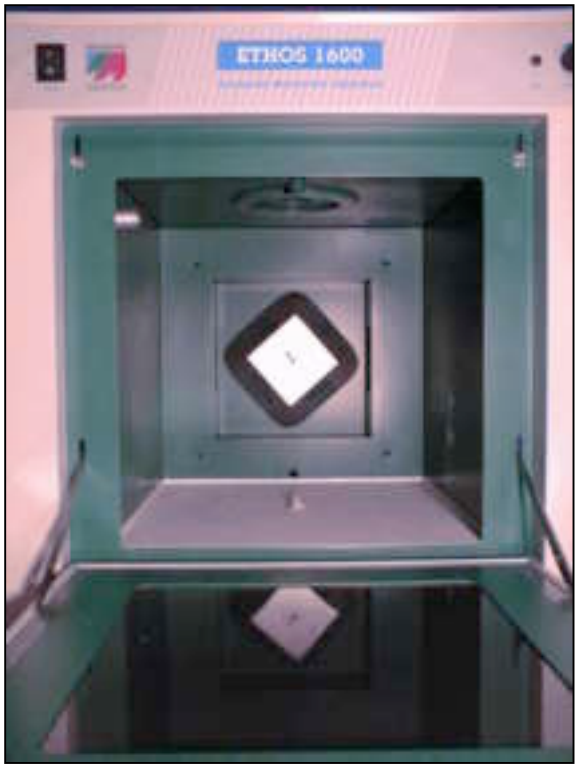
By monitoring the presence of NO_X in the microwave cavity (optional device), when the preset NO_X limits are exceeded, the microwave power is automatically reduced, triggering a "beep" signal.
Microwave emission increases again after the NO_X concentration has returned to the preset limits.
Volatile losses are prevented resulting in safer working conditions and more consistent results.

1.2.6 PERFORMANCE THROUGH MICROWAVE HARDWARE DESIGN

The “heart” of the ETHOS platform is the innovative and patented microwave feed/distribution system to the working cavity.
Two 800Watt industrial magnetron (ETHOS 1600 only), each with its own high voltage power supply, assure a powerful 1.600Watt of microwave installed power (picture).



Two high flow cooling fans guarantee peak performance of the magnetrons.
The microwaves are first fed into a “pre-mixing chamber.
From here an ingenious “pyramid shaped” rotating metal diffuser evenly distributes the microwaves in the cavity (picture).



Surfaces are Teflon coated with the diffuser protected by an inert high density polypropylene plate.
This unique microwave design and the advanced microprocessor control assures optimal microwave distribution in the cavity, thus preventing “hot spots”.
The installed power of 1.600Watt has been limited by design to operate at approximately 2/3 of the maximum power (1.000Watt) to assure longer magnetrons lifetime.
The 1.000Watt of delivered power are delivered in 10Watt increments to fit all types of applications.
The ETHOS 900 platform is powered by a 900Watt magnetron with a specially designed static diffuser to guarantee optimal microwave distribution in the cavity.

1.2.7 PERFORMANCE THROUGH VERSATILITY

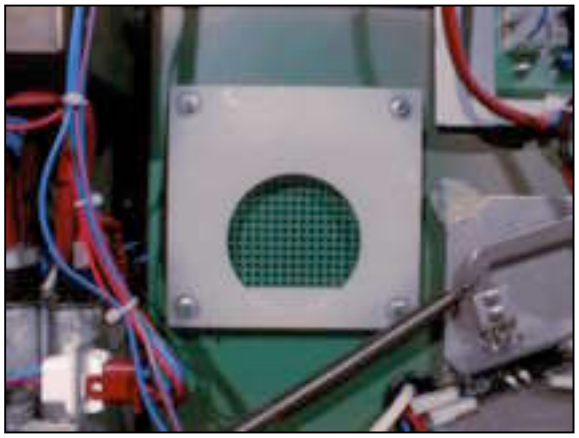
To fulfill the goals of making the ETHOS the most versatile microwave platform, Milestone's engineers built 13 microwave leakage protected (inlet/outlet) connectors.
Temperature and pressure sensor, connectors for vacuum and inert gas supply, can be all easily installed without any hardware modifications.

1.2.8 PERFORMANCE THROUGH MODULARITY

The ETHOS microwave platform has been designed with the microwave emission into the cavity from the back wall instead of the conventional position on top of the cavity.
This unique construction allows both the top and the bottom of the cavity to be free.
A free top allows the installation of batch reactors, while the free bottom makes possible the installation of a continuous flow reactor or of an advanced magnetic stirring device.
The ETHOS microwave platform can be upgraded with suitable modules/accessories to carry out: acid digestion, vacuum drying, evaporation/concentration, protein/peptide hydrolysis, solvent extraction, batch organic synthesis and continuous flow organic synthesis.

1.2.9 DURABILITY THROUGH CORROSION RESISTANCE

One of the key issues was assuring a long lifetime of our unit when performing digestions with aggressive acids (HNO_3, H_2SO_4, HCl, H_3PO_4, HF).
The all stainless steel hardware undergoes a pretreatment (sandblasting plus heat cycles) to assure best "bonding" conditions between the Teflon coating and metal surfaces.
The microwave cavity, the top cavity, the door and even the external surface of the air inlet (grid) are plasma coated with 5 layers of Teflon applied at 350°C (picture).



1.2.10 DURABILITY THROUGH CHASSIS DESIGN

The cabinet chassis is manufactured with magnetic 18/8 stainless steel (1,0mm gauge) to act as a Faraday cage to eliminate potential interference to other instruments (picture).

It is protected against acid/organic solvent vapors through high temperature polymer coating on both the outside and inside surfaces.

1.2.11 DURABILITY THROUGH ADVANCED EXHAUST/COOLING SYSTEM

This is another important factor for assuring long life of digestion vessels and safe working conditions.
The ETHOS platform is supplied with one (two up on request) heavy duty, $5m^3$/minute exhaust fan placed above the cavity and installed separately from the electronics to eliminate the danger of acid corrosion of the electronics (previous picture).
The air flow from the grids at the bottom of the cavity cools the external surfaces of the rotor and carries away any acid vapors released in the cavity.
An acid resistant flexible hose connects the exhaust fan to a fume hood assuring a safe working environment.

1.2.12 DURABILITY THROUGH USER-FRIENDLY SERVICE

As perfection is not of this world, easy access in case of servicing is a must.
Please refer to the separate ETHOS Service Manual for further information.

1.3 ETHOS 900 SAFETY AND MONITORING CONTROL OPTIONS

The ETHOS 900 microwave labstation can be fitted with any of the safety and monitoring control options below.

P/N	Description
33684	IRTC-500 Infrared temperature control
86300	APC-55E Automatic pressure control
33981	ATC-300 Automatic temperature control without sensor
33962	Temperature sensor model MW1Au gold plated
30009	SoftWAVE Method Development rev. 2.0 (requires PC, not supplied)

1.4 ETHOS 900 COMPATIBILITY WITH DIGESTION, DRYING AND CONCENTRATION ROTORS

The ETHOS 900 can accomodate the following acid digestion vessels and rotors:

P/N	Description
41400	AC-10 Advanced Carousel complete with 10 Vessels and Shields
34181	MDR-1000/3 complete
35301	MDR-1000/6S complete
41100	MDR-300/10S complete with PFA vessels
41200	MDR-300/10S complete with TFM vessels
35100	HPR-1000/10S rotor complete
35180	MPR-300/12S Rotor complete

The ETHOS 900 can accomodate the following drying and evaporation/concentration rotors:

P/N	Description
34670	MCR-1 Drying rotor in polypropylene 3,5L
40103	MCR-1 Drying rotor in polypropylene 6,5L
34649	MCR-6 Concentration rotor 6 position empty
34940	MCR-8 Concentration rotor 8 position empty
34510	MCR-12 Concentration rotor 12 position empty

The MCR series rotors require a vacuum source. Below are the options available.

P/N	Description
40018	FAM-40 Fume absorbing module 220V/50Hz
40019	FAM-40 Fume absorbing module 115V/60Hz
84245	VAC-20P Vacuum pump system 220V/50Hz
84254	VAC-60 Vacuum pump system 220V/50Hz

1.5 ETHOS 900 INSTALLATION

WARNING
Never install the ETHOS 900 under a fume hood!

In fact, if you should install the microwave system under a fume hood, the acid vapors might contaminate the air used for ventilating the electronic components, exposing them to a possible corrosion.

1.5.1 UNPACKING AND CHECKING LIST

When unpacking, verify that all parts correspond to the packing list included in the shipment.
Utmost care must be taken during unpacking of the ETHOS 900 to avoid any scratch or damage to the special acid and organic solvent resistant external coating.

Carefully cut the protective belts, as shown in the next picture.

Remove the carton box located in the upper part of the box (picture) and keep it apart.
It contains accessories which will be taken during the installation of the ETHOS 900.
The carton box should include the following:

- n. 1 Milestone ETHOS 900 microwave labstation;
- n. 1 Power cord for ETHOS 900;
- n. 1 Plastic hose 2m long;
- n. 1 Control terminal model “240”;
- n. 1 Interface cable 5-pin for connecting the ETHOS 900 to the control terminal model “240”.

Lift-up the main carton box to facilitate the removal of the ETHOS 900 (picture).
The ETHOS 900 comes protected by a plastic bag to avoid problems of wetting during its transportation.

Provide a bench of at least 1m length and 80cm wide and locate the ETHOS 900 on the bench.
A proper location of the ETHOS 900 is next to a fume hood, since the microwave unit should be connected to the fume hood by using the hose supplied (details will follow).

WARNING
The weight of the ETHOS 900 is approximately 85kg. At least 3 persons should remove the system from its box and locate it on the bench.

Open now the boxes containing the accessories of the ETHOS 900.

Notice the ETHOS 900 power cord (picture).

Notice the plastic hose 2ml long (picture).

Notice also the control terminal model “240” with its interface cable 50 pins (picture).

1.5.2 ETHOS 900 OVERVIEW

Notice some of the most important details of the ETHOS 900.
Main swicth (picture).

WARNING
During the ETHOS 900 installation the main switch should be in the “OFF” position.

Exhaust module main switches (picture).

The ETHOS 900 comes with one exhaust module as standard feauture.
Optionally it is possible to install a second exhaust module.
The “VENT 1” swicth refers to the exhaust module built-in as standard feature, while the “VENT 2” swicth refer to the second optional exhaust module.

WARNING
During the ETHOS 900 installation both the switches should be in the “OFF” position.

Note the two openings at the side of the ETHOS 900 (picture).

Those are the air inlets for the air exhaust system, located in the upper part of the system.
Not to be worry about microwave leakages!
A metal grit, which can be seen through the plastic connection, does reflect microwaves back to the cavity, not allowing any leak through the opening.
Do not block or obstruct the openings, since air circulation trough the microwave cavity is an important safety aspect of microwave closed vessel acid digestion.
Overiew of the back side of the ETHOS 900 (picture).

Notice in the upper part (left) the open connection for the plastic hose and at right the second connection (closed) for a second otional blower.
Detail of the interfaces of the ETHOS 900 (picture); at left a standard RS-232 9 pin interface port, to be used for connecting the microwave to external devices; at righ the special 50 pin interface port to be used for connecting the ETHOS 900 to the control terminal model “240”.

Detail of the left panel of the ETHOS 900 (picture), showing the connection to the power cord (at right, down), the power connection to external devices (right, up), and two fuses.

More details about this subject are available in the ETHOS Service Manual.

1.5.3 CONTROL TERMINAL “240” INTERFACE PORTS

The picture below shows the back side of the control terminal model “240”.
From left to right it is possible to see the following interface ports:

- RS-232 9 pin for connecting the ETHOS 900 to an external PC;
- RS-232 9 pin for connecting the ETHOS 900 to an external balance (a limited number of balances can be interfaced);
- special 50 pin interface port for connecting the control terminal “240” to the ETHOS 900 main unit;
- Centronix interface for connecting the ETHOS 900 to any printer (parallel port);
- analogue port for connecting the ETHOS 900 to an external laboratory recorder.

1.5.4 ETHOS 900 SETUP

Connect the 50 pin interface cable supplied to the back side port of the ETHOS 900 and use a screwdriver to fix them well (picture).

Connect the other end of the 50 pin cable to the control terminal “240” and again use a screwdriver to fix them well (picture).

WARNING
Both the 50 pin connections must be very tight; damages may occour when the ETHOS 900 and the control terminal “240” are not properly connected.

Plug-in the supplied power cord at the back side of the ETHOS 900 (picture).

Introduce now the “L” shaped adapter to the exhaust outlet located at the left of the ETHOS 900 back panel.
Connect the exhaust hose to the “L” shaped adapter (picture).

The free end of the exhaust hose should be placed under a fume hood or directly outside the laboratory (picture).

Your new ETHOS 900 is now ready to work!

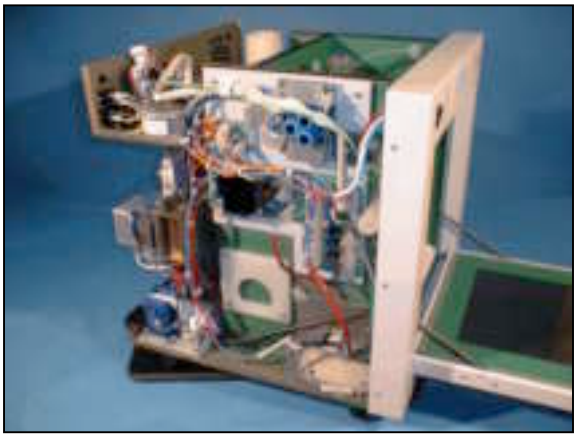
A LOOK AT THE HIDDEN SIDE OF THE ETHOS UNIT

The most important safety features are the ones you don't see from the outside.
For this reasons Milestone lets you see under the cabinet cover of the ETHOS.

SAFETY THROUGH MECHANICAL DESIGN

The ETHOS is not a kitchen type nor a commercially modified kitchen microwave unit; it is a rugged, heavy duty professional system designed to guarantee years and years of trouble free and safe operation.
The microwave cavity is quite large, to accommodate different types of accessories.
Dimensions are 35cm x 35cm x 35cm, for a volume of approximately 43L.

As a standard feature, the door is made of stainless steel; however, on request (ETHOS 1600 only), it is possible to install a door with a high impact shock resistant 27cm x 27cm glass window for easy viewing of the illuminated cavity (picture).The hardware is manufactured with 18/8 stainless steel, 1.5mm gauge (picture).



SAFETY THROUGH PATENTED DOOR DESIGN

The door design is the most critical issue when developing new microwave hardware.
Mechanical strength and perfect sealing of the cavity to avoid microwave leaks are prerequisites.
Milestone's engineers, on the basis of the "movable door" principle used in high pressure autoclaves, designed a door with an innovative opening/self resealing mechanism.
The door frame is mounted on four spring-loaded high strength steel bars (picture).

In the event of a sudden overpressure inside the cavity, the entire door opens horizontally ("moves out") presenting a large surface area for immediate and safe release of the overpressure. Microwave power is instantaneously cut off. Immediately afterward, the door is "pulled back" by the springs, resealing the cavity.

The microwave cavity door opens downwards to a flat horizontal position, forming a stable work platform for handling microwave accessories before and after digestion procedures.

SAFETY TROUGH ELECTRICAL DESIGN

The ETHOS microwave platform is equipped with a total of 10 microswitches to assure the highest safety conditions. Four (two on each side of the door) fast release microswitches instantaneously interrupt the microwave emission whenever the door is opened (previous picture).
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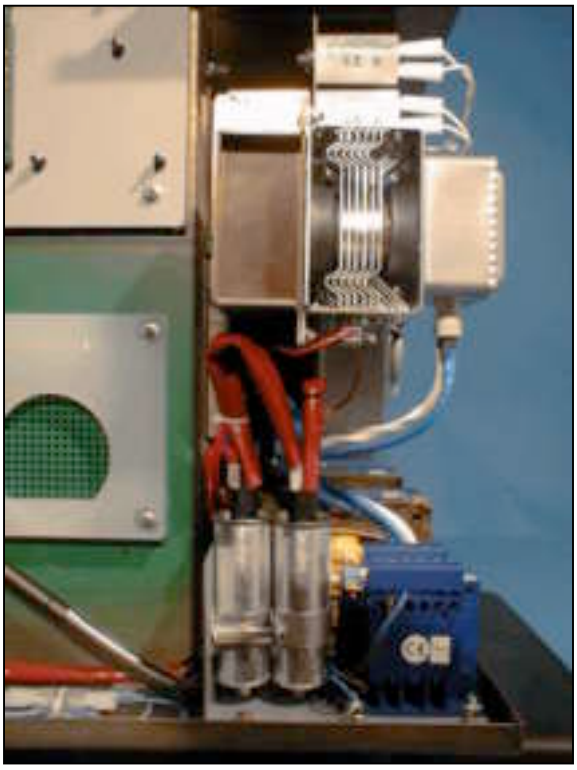

SAFETY THROUGH NO_X MONITORING

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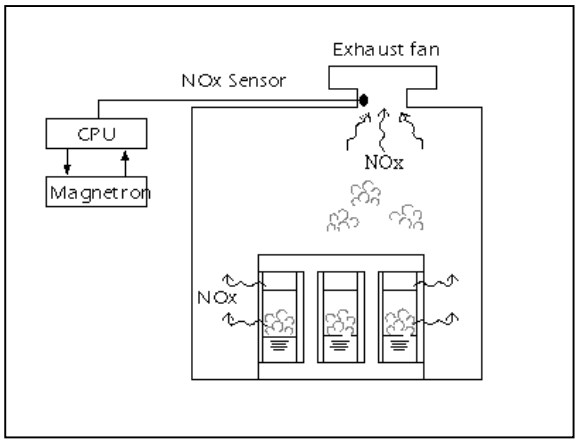
NO_X gases may diffuse through the vessel walls or are released during vessel overpressurization, in the microwave cavity (picture).



By monitoring the presence of NO_X in the microwave cavity (optional device), when the preset NO_X limits are exceeded, the microwave power is automatically reduced, triggering a “beep” signal.
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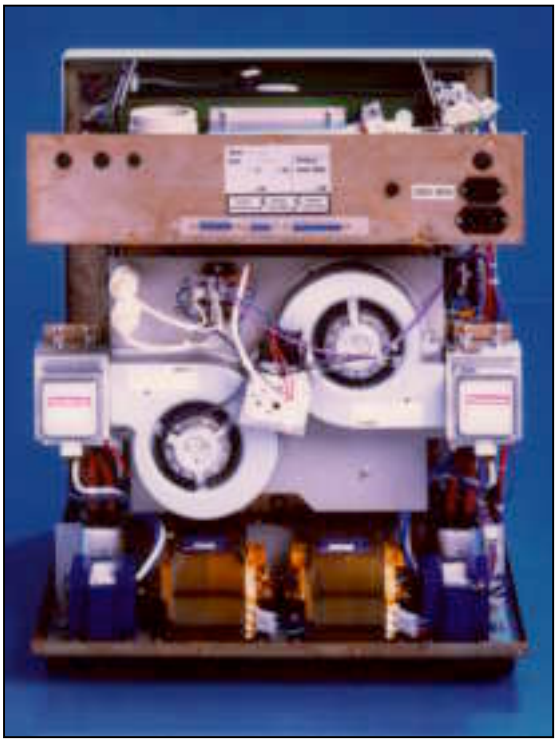

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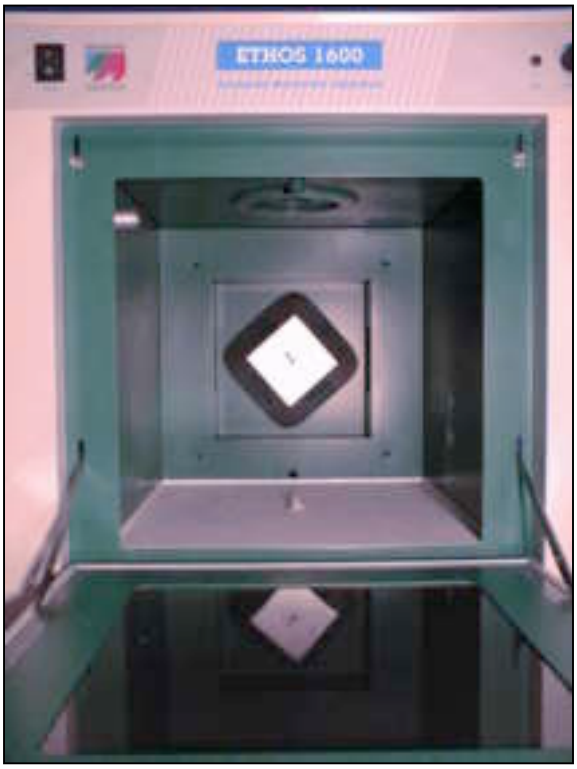

Surfaces are Teflon coated, with the diffuser protected by an inert high density polypropylene plate.
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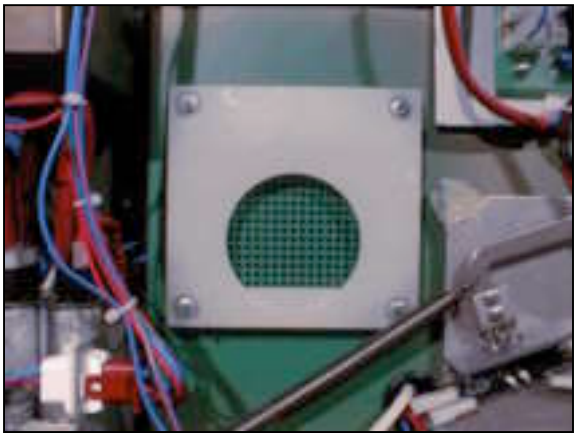

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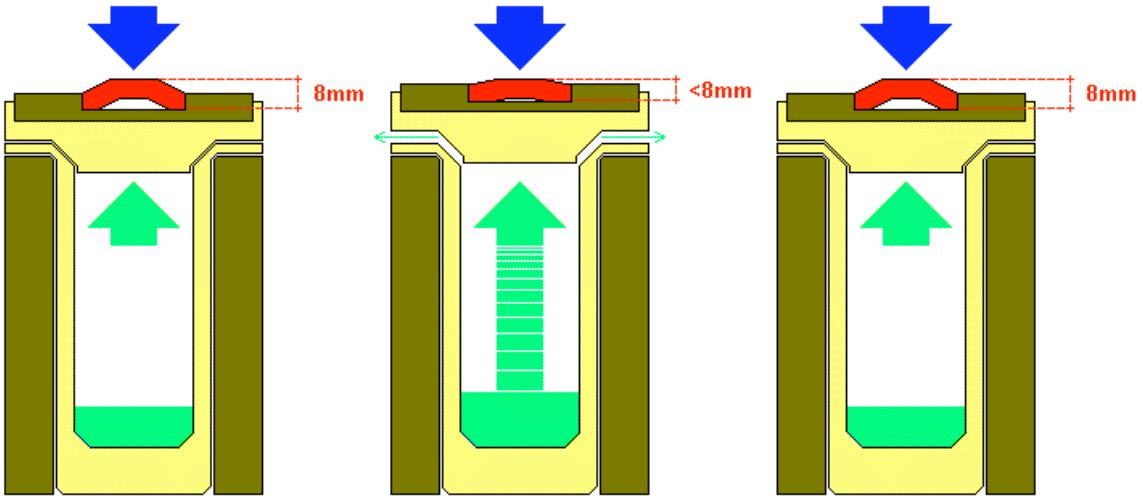
33981	ATC-300 Automatic temperature control without sensor
33962	Temperature sensor model MW1Au gold plated
30009	SoftWAVE Method Development rev. 2.0 (requires PC, not supplied)

MDR TECHNOLOGY

Milestone patented microwave digestion rotor (MDR) technology provides analysts with unsurpassed performance capabilities and the highest standard of safety in closed vessel microwave digestion.
MDR rotors hold multiple microwave digestion vessels in a high strength one piece “monobloc” rotor body.
This unique design securely retains sample vessels during microwave digestion and subsequent venting of decomposition gases, for maximum user safety.
The digestion vessels used in MDR rotors have no molded or machined threads to be chemically attacked and weakened by acid vapors during vessel venting and opening.
This unique feature is an important factor in the durability and safety of Milestone digestion vessels.
All digestion vessels in MDR rotors are protected by a patented reclosing (vent and reseal) relief valve mechanism.
Vessels fitted into a MDR rotor body are sealed for microwave digestion and proper relief valve functioning using a calibrated torque wrench.
The torque wrench ensures vessels are sealed for maximum operating pressure, and will safely vent when a buildup of decomposition gases in excess of vessel’s maximum operating pressure is reached.
Microwave digestion of organic samples frequently involves exothermic reactions which instantaneously generate large amount of decomposition gasese (CO_2 and NO_X) as the sample is oxidized.
For proper overpressurization protection of microwave digestion vessels, the venting capacity of the relief device must be equal or higher than the rate of decomposition gas formation.
Only Milestone’s relief valve is capable of instantaneously venting excess decomposition gases and resealing the vessel for completation of the digestion procedure.
The digested sample is retained inside the vessel for analysis.

MDR SAFETY



The above drawing clearly show the instantaneous release of overpressure due to the deformation of the HTC safety spring.
The figure at left represents a vessel in normal working conditions.
When pressure increases over the maximum spring working pressure (figure in the center), the entire cover is allowed to lift up because of the elastic deformation of the safety spring.
The overpressure is instantaneously released thanks to the large surface area.
The larger the surface area the shorter the time required to release the pressure.
Once the pressure inside the vessel has returned within the vessel limit, the cover reseal the vessel, permitting the operator to continue with the acid digestion without any loss (figure at right).

This is the unique operating principle of the microwave digestion rotor technology of Milestone.
An indicator ring located next to the cover remains in the upper position in case of vessel venting.
The picture shows at left a vessel in normal conditions and at right a vessel after venting.

GUIDELINES FOR MICROWAVE ACID DIGESTION

INTRODUCTION TO THE REAGENTS MOST COMMONLY USED IN MICROWAVE DIGESTION

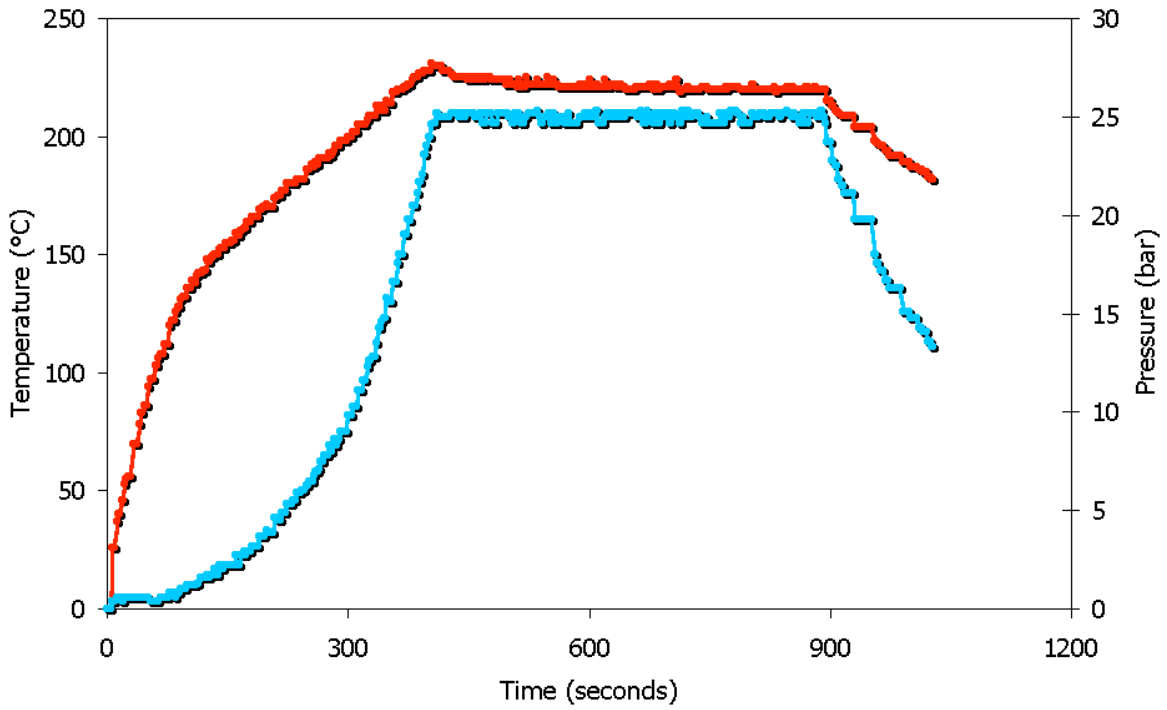
The acids used in microwave digestion may be classified in two main groups:

- nonoxidizing acids, such as hydrochloric acid, hydrofluoric acid, phosphoric acid, diluted sulfuric acid and diluted perchloric acid;
- oxidizing acids, such as nitric acid, hot concentrated perchloric acid, concentrated sulfuric acid and hydrogen peroxide.

NITRIC ACID

Nitric acid has the following properties:

- boiling point is 120°C at 65% concentration;
- poor oxidizing strength at concentrations less than 2M; oxidizing strength increases with concentration and reaction temperature;
- most common acid for oxidation of organic matrices with this typical reaction: $(CH_2)_X + 2HNO_3 \rightarrow CO_2(g) + 2NO + 2H_2O$;
- it dissolves most metals forming soluble nitrates, exceptions are Au and Pt (not oxidated) and Al, B, Cr, Ti and Zr (passivated);
- these metals require acid mixtures or diluted nitric acid;
- often mixed with H_2O_2, HCl and H_2SO_4;
- available in high purity for trace level analysis.



The above graphic represents the temperature and pressure profile of nitric acid up on microwave heating. Notice that pressure has been controlled at 25bar, resulting in a temperature of 225°C.

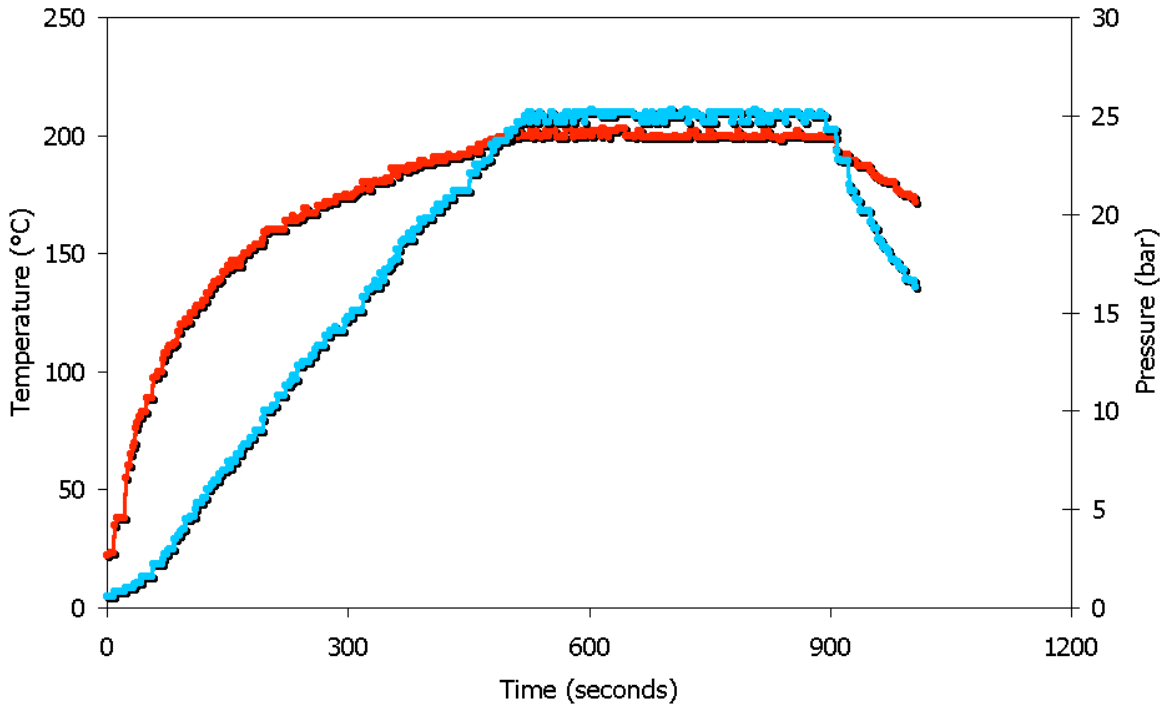
HYDROGEN PEROXIDE

Hydrogen peroxide is an oxidizing agent ($2H_2O_2 \rightarrow 2H_2O + O_2$); added to nitric acid it reduces the nitrous vapours and it accelerates the digestion of organic samples by raising the temperature.
A typical mixture ratio is HNO_3:H_2O_2= 4:1 (volume/volume).

HYDROCHLORIC ACID

Hydrochloric acid has the following properties:

- boiling point of azeotropic mixture with H_2O with 20,4% HCl is 110°C;
- available with 38% concentration;
- it dissolves salts of weak acids (carbonates, phosphates) and most metals are soluble with the exception of AgCl, HgCl and TiCl;
- excess of HCl improves the solubility of AgCl, converted into $AgCl_2^-$;
- strong complexing nature;
- widely used for iron-based alloys because of its ability to hold large amounts of chloro-complex in solution;
- other complexes formed are Ag (I), Au (II), Hg (II), Ga (III), Tl (III), Sn (IV), Fe (II) and Fe (III);
- it does not dissolve oxides of Al, Be, Cr, Ti, Zr, Sn and Sb; sulphates of Ba and Pb, group II fluorides, SiO_2, TiO_2 and ZrO_2.



The graphic above represents the temperature and pressure profile of hydrochloric acid up on microwave heating. Notice that pressure has been controlled at 25bar, resulting in a temperature of 205°C.

HYDROFLUORIC ACID

Hydrochloric acid has the following properties:

ACID DIGESTION

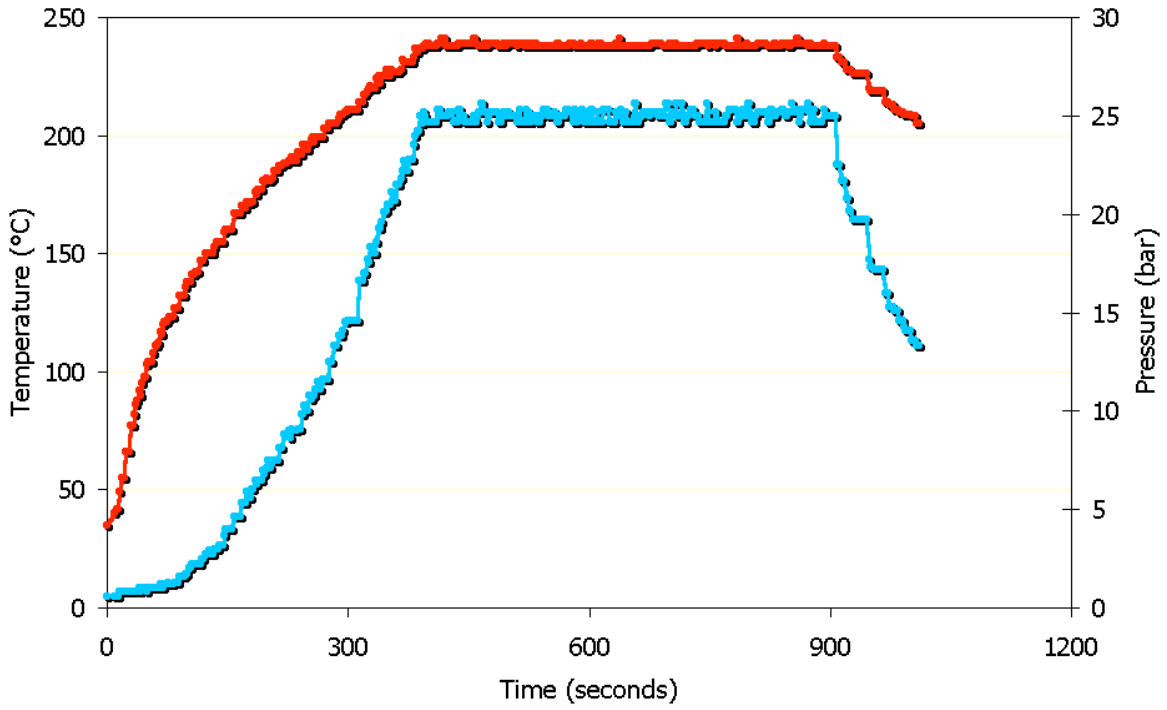
- Boiling point is 108°C at 40% concentration;
- nonoxidizing, strong complexing nature;
- used in digestion of minerals, ores, soils, rocks and even vegetables containing silicates;
- major use is the decomposition of silicates according with this reaction: $SiO_2 + 6HF \rightarrow H_2SiF_6 + 2H_2O$;
- often used in combination with HNO_3 or $HClO_4$.

EVAPORATION/CONCENTRATION

- Following dissolution, many analyses require removal of HF to prevent equipment damage or to resolubilize insoluble fluorides;
- many analytes such as As, B, Se, Sb, Hg and Cr may volatilize.

COMPLEXATION

- Alternative approach to remove HF from the solution, by addition of Boric acid;
- the following reactions take place: $H_3BO_3 + 3HF \rightarrow HBF_3(OH) + 2H_2O$ and $HBF_3(OH) + HF \rightarrow HBF_3 + H2O$;
- 10-50 times excess Boric acid enhances reaction rate.

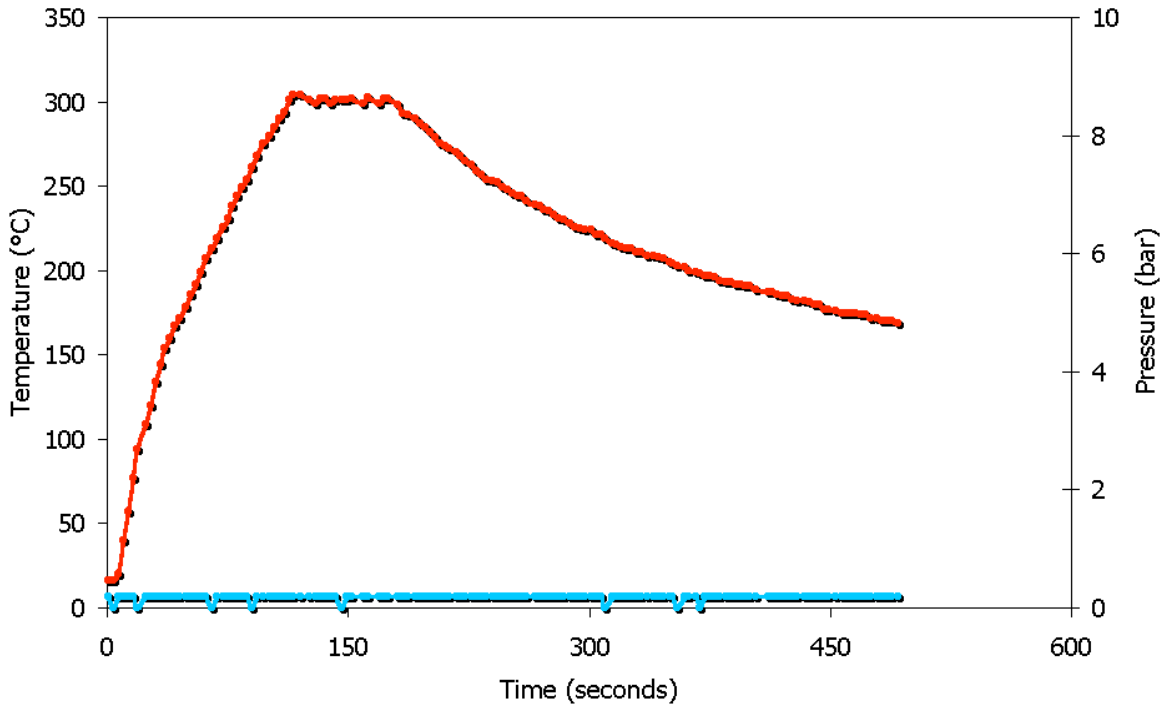


The above graphic represents the temperature and pressure profile of hydrofluoric acid up on microwave heating. Notice that pressure has been controlled at 25bar, resulting in a temperature of 240°C.

SULFURIC ACID

Sulfuric acid has the following properties:

- boiling point is 340°C at 98% concentration, exceeding the maximum working temperature of TFM Teflon vessels;
- careful reaction monitoring is required to prevent vessel damages;
- it destroys organics by dehydrating action;
- many sulfates are insoluble (Ba, Sr, Pb).



The above graphic represents the temperature and pressure profile of sulfuric acid up on microwave heating.
Notice that temperature has been controlled at 300°C (for 1 minute only), without any pressure increase.
It is clear that the use of concentrated sulfuric acid in the MDR rotors poses significant problems because of its high boiling point.
300°C are critical for TFM Teflon vessels and simply too high for PFA Teflon vessels (they melt down at such temperature).
It is therefore advisable to use sulfuric acid only with a strict temperature control.

1.0 MDR TECHNOLOGY

Milestone patented microwave digestion rotor (MDR) technology provides analysts with unsurpassed performance capabilities and the highest standard of safety in closed vessel microwave digestion.
MDR rotors hold multiple microwave digestion vessels in a high strength one piece “monobloc” rotor body.
This unique design securely retains sample vessels during microwave digestion and subsequent venting of decomposition gases, for maximum user safety.
The digestion vessels used in MDR rotors have no molded or machined threads to be chemically attacked and weakened by acid vapors during vessel venting and opening.
This unique feature is an important factor in the durability and safety of Milestone digestion vessels.
All digestion vessels in MDR rotors are protected by a patented reclosing (vent and reseal) relief valve mechanism.
Vessels fitted into a MDR rotor body are sealed for microwave digestion and proper relief valve functioning using a calibrated torque wrench.
The torque wrench ensures vessels are sealed for maximum operating pressure, and will safely vent when a buildup of decomposition gases in excess of vessel’s maximum operating pressure is reached.
Microwave digestion of organic samples frequently involves exothermic reactions which instantaneously generate large amount of decomposition gasese (CO_2 and NO_X) as the sample is oxidized.
For proper overpressurization protection of microwave digestion vessels, the venting capacity of the relief device must be equal or higher than the rate of decomposition gas formation.
Only Milestone’s relief valve is capable of instantaneously venting excess decomposition gases and resealing the vessel for completation of the digestion procedure.
The digested sample is retained inside the vessel for analysis.

1.1 MONOBLOC MDR ROTORS

Rotor	MDR-300/10S
P/N	41100
Vessels	10
Vessel material	PFA Teflon
Maximum pressure	30bar
Maximum temperature	240°C

Rotor	MDR-300/10S
P/N	41200
Vessels	10
Vessel material	TFM Teflon
Maximum pressure	30bar
Maximum temperature	240°C

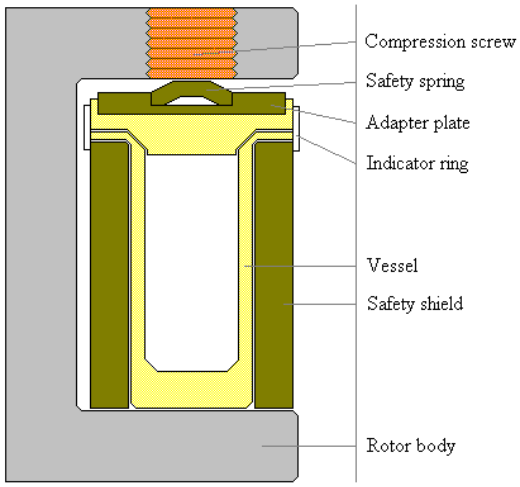
Rotor	MDR-1000/6S
P/N	35301
Vessels	6
Vessel material	TFM Teflon
Maximum pressure	100bar
Maximum temperature	300°C

1.2 SEGMENTED MDR ROTORS

Rotor	MPR-300/12S
P/N	35180
Vessels	12
Vessel material	PFA Teflon
Maximum pressure	30bar
Maximum temperature	240°C

Rotor	HPR-1000/10S
P/N	35100
Vessels	10
Vessel material	TFM Teflon
Maximum pressure	100bar
Maximum temperature	300°C

1.3 MDR MAJOR COMPONENTS



The rotor consists of a high density polypropylene core in which a number of niches have been "carved" along the periphery by a numerical control machining center.
The compact core has the required strength to withstand the extreme pressure generated inside the vessel during digestion.
The core tolerates this pressure without any deformation and therefore with high safety.
Special plastic compression screws are located on the top of the rotor next to each "niche".
Suitable pressure is applied to the dome-shaped spring holding the cover to the top of the vessel by tightening the screws to the appropriate degree.
The dome-shaped, flat-top spring is the "heart" of the MDR technology and is made of HTC, a new high-performance plastic.
HTC is crystalline and can act as a powerful and very reliable spring.
The adapter plate is placed on top of the cover.
Made of a plastic with high resistance to mechanical compression, it allows a very homogeneous and repeatable distribution of the preset pressure limit.
The cover has a flat bottom rim and perfectly fits the top rim of the vessel.
Sealing between cover and vessel takes place on a very large surface area without any intermediate seal ring which could deteriorate in contact with acid vapors.
The elimination of a threaded cover prevents all problems connected with thread deformation or impurities on threads.
The vessel has normally a volume of 100mL.
A high resistant HTC safety shield prevent lateral deformation of the vessel.

1.4 MATERIALS USED FOR THE MANUFACTURE OF MDR VESSELS AND SAFETY SHIELD

1.4.1 TFM TEFLON

TFM is often used for the manufacture of covers and vessels of the MDR rotors.
TFM is a Hoechst trade name for chemically modified PTFE.
TFM Teflon has a very dense structure with smooth surface and low permeability to gases and vapors.
TFM Teflon is chemically inert to most mineral acids and combinations thereof.
It is a thermal insulator, with a very high stability to temperature extremes.
TFM Teflon is microwave transparent.
Its maximum working temperature for extended use is 260°C, for short time 300°C.
The melting range is 320-340°C.

1.4.2 PFA TEFLON

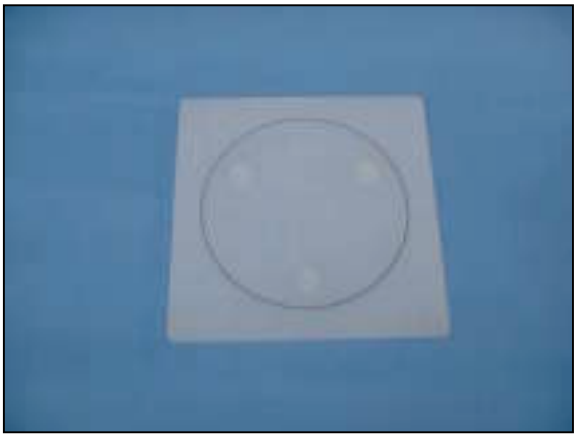
PFA is often used for the manufacture of vessels of the MDR rotors.
PFA Teflon is a melt-processable fluoroplastic material which can be extruded and therefore manufactured in relatively low-cost vessels.
It shows a good all-round chemical resistance.
It is microwave transparent and termal insulator.
The maximum working temperature is up to 260°C; the melting temperature is about 300°C.

1.4.3 HTC

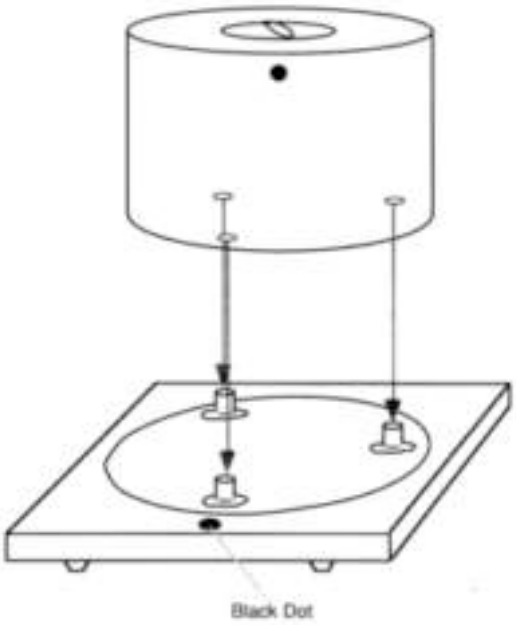
HTC plastics are used for the manufacture of safety spring, adapter plate and safety shield.
They are characterized by the sequence ether-ether-keton-keton.
When HTC are used for the safety shield they are reinforced with glass fiber.
HTC have high mechanical strength to high temperatures and good chemical resistance.
The maximum service temperature for extended use is 250°C.

1.5 ACCESSORIES OF THE MDR MONOBLOC ROTORS

1.5.1 WORK STATION



The work station (P/N 34030) allows fixing of every kind of monobloc rotors in order to properly close and open the vessels (drawing).



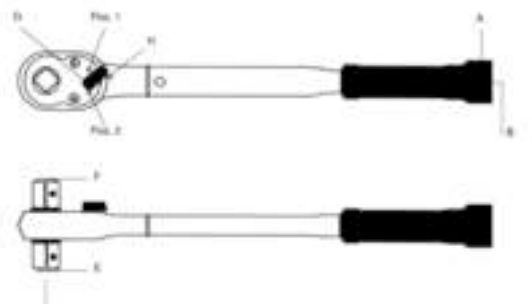
The work station is made of polypropylene and it is provided with four anti-skid rubber feet.

1.5.2 TORQUE WRENCH

The torque wrench (P/N 34000) is used to properly and safely close/open the vessels of the MDR rotors.

1.5.3 INSTRUCTION OF USE

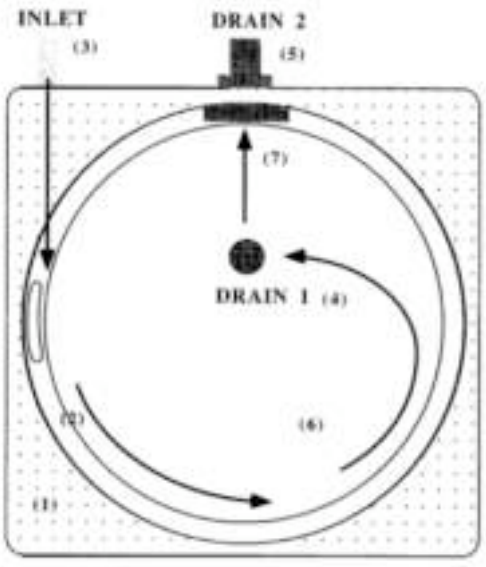
Apply a smooth steady pull until you hear a click. Release your pressure on the handle and the torque wrench will automatically reset itself provided the ratchet reverse lever G is in position 1 (toward the indicator H).
Refer to the drawing above.



The torque function does work only when the reverse lever G is in position 1.
By placing it in position 2 the torque function is neutralized, the the wrench becomes a normal reversible ratchet.
The torque is factory-set at 22,5Nm for all MDR rotors.
Note that for correct torque setting, the tool should be gripped properly on the middle of the handle and not on the selection knurl A.

1.5.4 COOLING SYSTEM

The cooling system (P/N 40014) can accomodate every kind of monobloc rotors.
Its design allows a continuous tangential water circulation and exchange inside the container, therefore permitting a very rapid cooling of the rotor.
The complete cooling system includes the following items (drawing).



- Polypropylene bottom plate (1);
- Plexiglass transparent container (2);
- Water inlet complete with junction (3);
- Water outlet for drain (4);
- Outlet junction for drain (5);
- Plexiglass protection cover (6);
- Overflow control device (7).

1.5.5 COOLING SYSTEM INSTALLATION AND OPERATION

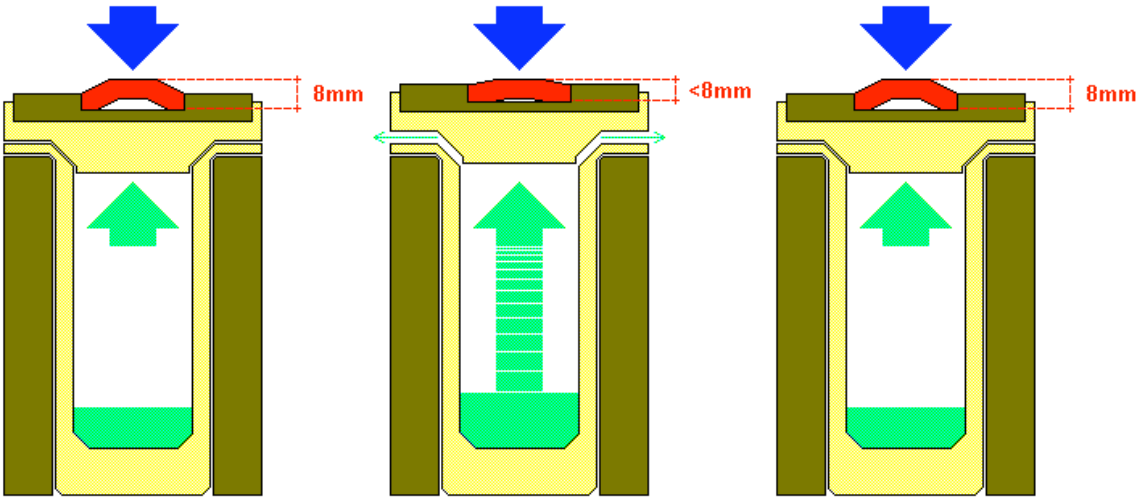
To install the cooling system, simply follow the procedure below.

1. Unpack the cooling system.
2. Connect it to the water supply (junction diameter is 1/2").
3. Place the cooling system near the ETHOS 900/1600 and next to a water supply and a water drain.
4. Connect with rubber hose the inlet (3) to the water supply which must be provided with a suitable valve.
5. Connect the drain (5) to the laboratory drain.

The cooling system is ready for operation.
After the microwave digestion, take the rotor out of the unit and place it inside the cooling tank, after removing the plexiglass cover of the system.
Relocate the plexiglass cover in its position.
The bottom of the rotor closes the drain 1 at the bottom of the tank.
Carefully supply water and see that water slowly fills up the tank until the maximum level (overflow) is reached.
Adjust, if required, the water flow by using the valve of the water supply.
Let the rotor cool for at least 10 minutes; during this time any water surplus will be drained by the overflow.
Check that the water is circulating around the rotor and that the level is below the indicator rings of the vessels by about 10mm.
Close the water inlet.
Take away the plexiglass cover and remove the rotor.
The drain 1 opens and in few seconds the tank will be completely empty.
Dry the rotor body with a tissue and place it on the work station.

1.6 MDR SAFETY

The larger the surface area the shorter the time



The above drawing clearly show the instantaneous release of overpressure due to the deformation of the HTC safety spring.
The figure at left represents a vessel in normal working conditions.
When pressure increases over the maximum spring working pressure (figure in the center), the entire cover is allowed to lift up because of the elastic deformation of the safety spring.
The overpressure is instantaneously released thanks to the large surface area.
required to release the pressure.
Once the pressure inside the vessel has returned within the vessel limit, the cover reseal the vessel, permitting the operator to continue with the acid digestion without any loss (figure at right).
This is the unique operating principle of the microwave digestion rotor technology of Milestone.
An indicator ring located next to the cover remains in the upper position in case of vessel venting.
The picture belows shows a vessel in normal conditions and at right a vessel after venting.

1.7 MDR-300/10S

The MDR-300/10S medium pressure rotor contains 10 vessels for acid digestion of routine and moderately difficult samples at operating pressure up to 30bar (435psi).
The MDR-300/10S is available in two different versions: with PFA Teflon vessels or with TFM Teflon vessels.

- P/N 41100 MDR-300/10S with PFA Teflon vessels.
- P/N 41200 MDR-300/10S with TFM Teflon vessels.

1.7.1 MDR-300/10S CONFIGURATIONS

The MDR-300/10S rotor with PFA Teflon vessels has P/N 41100 and it is composed by the following items.

P/N	Quantity	Description
34642	1	Rotor body
34021	10	Safety spring
34114	10	Adapter plate
34115	10	TFM Teflon cover
34049	10	Indicator ring
34121	10	PFA Teflon vessel
34602	1	Safety shield (set of 10)

The MDR-300/10S rotor with TFM Teflon vessels has P/N 41200 and it is composed by the following items.

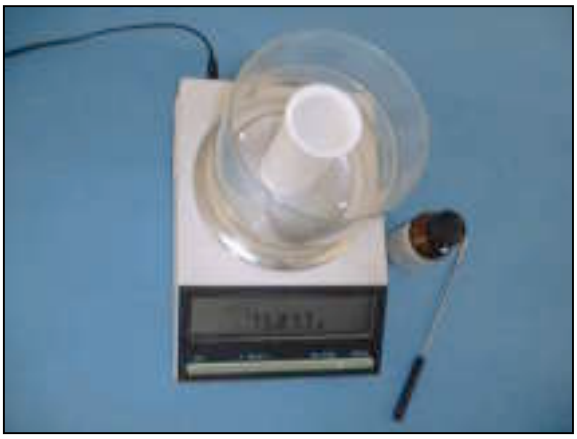
P/N	Quantity	Description
34642	1	Rotor body
34021	10	Safety spring
34114	10	Adapter plate
34115	10	TFM Teflon cover
34049	10	Indicator ring
33802	10	TFM Teflon vessel
34602	1	Safety shield (set of 10)

1.7.2 HOW TO OPERATE WITH THE MDR-300/10S ROTOR

The above procedure relates to the use of a MDR-300/10S with TFM Teflon vessels (P/N 41200). The same sequence can be followed when using a MDR-300/10S with PFA Teflon vessels (P/N 41100).

1.7.3 SAMPLE WEIGHING, REAGENTS ADDITION AND VESSEL CAPPING

Place a TFM Teflon vessel directly on the balance plate.
The weight of the TFM Teflon vessel is about 75g, therefore most analytical balances may be used for the pourpose.



Tare the vessel and directly weigh the sample by placing it inside the vessel.
Try to reduce as much as possible sample transfer, to avoid any contamination.

Introduce the TFM Teflon vessel into the HTC protection shield.

WARNING
The wall surfaces of the HTC protection shield as well as the outer wall of the TFM Teflon vessel must be dry and clean.
Damages may occour otherwise!

Add suitable reagents to the sample.
When part of the sample is left on the inner wall of the TFM Teflon vessel, try to wet it by adding acids drop by drop.

Gently swirl the solution, to homogenise the sample with the acid.
Place the TFM Teflon cover on the TFM Teflon vessel.
Push it by end and check that the cover fits well onto the vessel.

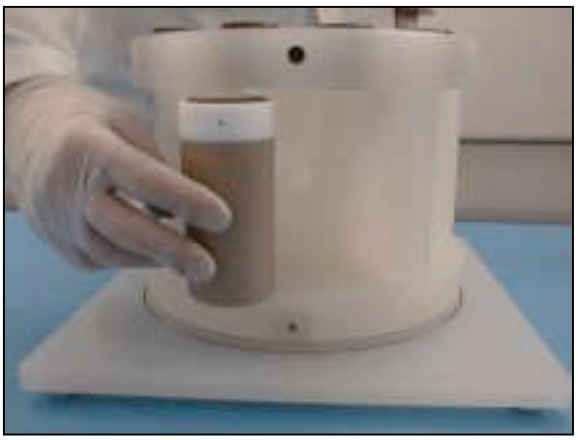
Place now the HTC adapter plate on the TFM Teflon cover, with the flat part of it at facing downwards, to have the space for the HTC safety spring on the top side (picture).

Locate the HTC safety spring onto the HTC adapter plate (picture).

Place the TFM Teflon indicator ring on the cover and push it down completely, until it fits the groove of the HTC safety shield, as shown by the next picture.

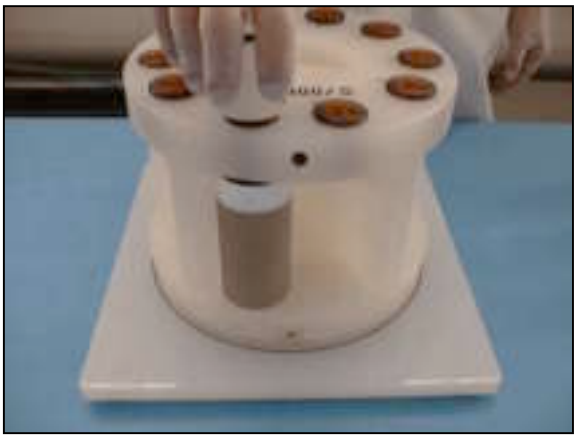
Position the polypropylene rotor body on the work station; use the black coloured dot on the rotor body as reference point to fit the rotor body on the work station (picture).

Introduce the vessel vertically into one of the numbered niches carved along the periphery of the 10-position polypropylene rotor body.
Notice the position of the venting hole located in the TFM Teflon indicator ring.
The hole must face outside, so that in case of venting no hot acid vapors can damage the polypropylene rotor body (picture).



It may be useful now to fix the vessel inside its location, without a complete closing.
Take the torque wrench and remove the plastic adaptor (picture).

Use the adaptor to fix the vessels, as shown in the next picture.



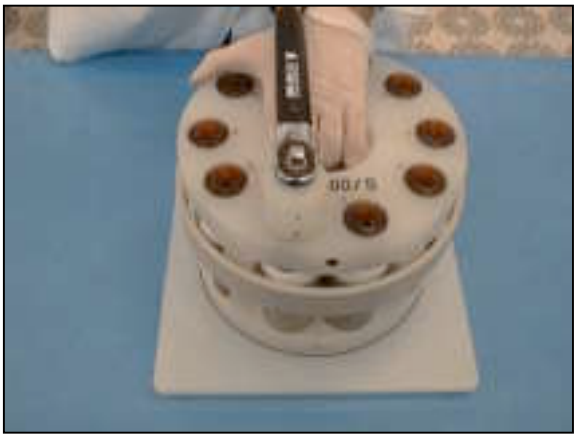
Repeat the same operations for the remaining nine vessels; then put the external protection ring around the rotor body (picture).

Take the torque wrench and set the indicator to the “CLOSE” position, as shown in the next picture.

Tighten the HTC screw in the upper part of the rotor body using the torque wrench.
Make long rotations (refer to the two next pictures) instead of short ones and do proceed slowly until you hear a clicking sound informing that the vessel is properly closed inside its niche.

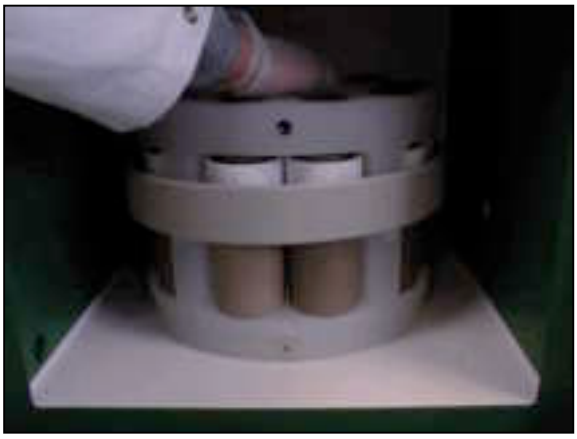
WARNING
Stop turning the torque wrench when the click is heard.
In fact the torque wrench may overload the HTC safety spring if further rotation is applied.

Make slow 90° rotations with the torque wrench, to get a better control of the correct closing of the vessel (click sound).



The MDR-300/10S rotor is now ready to be introduced inside the ETHOS 900/1600; open the door of the microwave unit by pressing down the door handle (picture).

Look at the position of the HTC drive adaptor of the ETHOS 900/1600.
Press “F7” on the control terminal keyboard to rotate the HTC drive adaptor until it reaches the position shown in the picture above, with the flat side facing the ETHOS 900/1600 door.



Slide-in the MDR-300/10S rotor inside the microwave cavity of the ETHOS 900/1600.
Notice the position of the black coloured dot on the polypropylene rotor body, facing the door of the microwave unit.
In this way the rotor is perfectly aligned with the HTC drive adaptor.

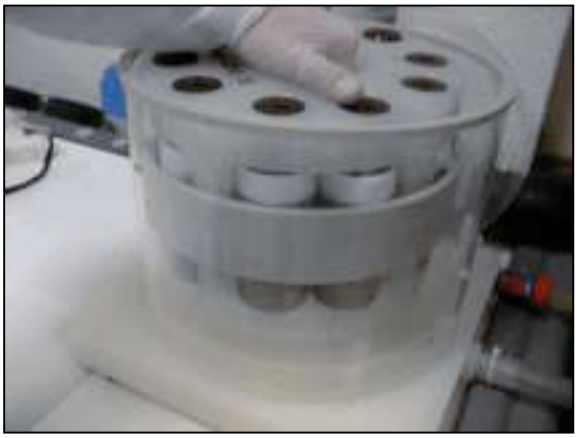
Close the door of the ETHOS 900/1600 (picture).

Switch on the ETHOS 900/1600 (picture).

Start a suitable microwave acid digestion program.

1.7.4 COOLING DOWN AND UNCAPPING THE VESSEL

Once the digestion program is completed, high temperature and pressure are reached inside the vessels, according with the sample preparation procedure applied.
It is therefore necessary to cool down the rotor before opening the vessels.
To facilitate this operating step Milestone has developed a cooling system able to reduce the cooling time to about 10 minutes.



Introduce the rotor body complete with vessels and protection ring inside the cooling system (picture above).

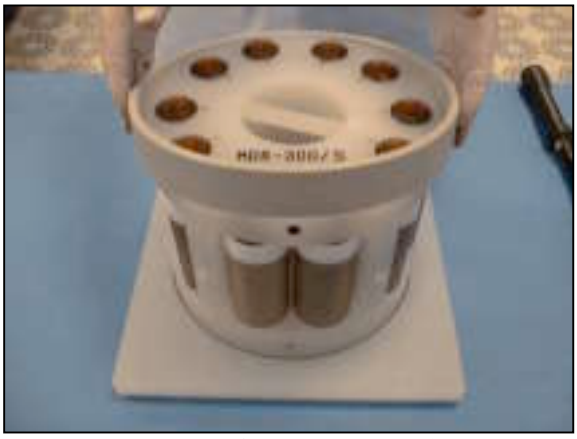
Put the cover back on the cooling system itself and turn on the (tap) water.

Let the (tap) water flow in for approximately 10-15 minutes.
Turn off the water supply and move the rotor body from the cooling system to the work station, preferably located under a fume hood.
Dry with the utmost care rotor and vessels prior uncapping.
Take the torque wrench and set the indicator to the "OPEN" position, as shown in the next picture.

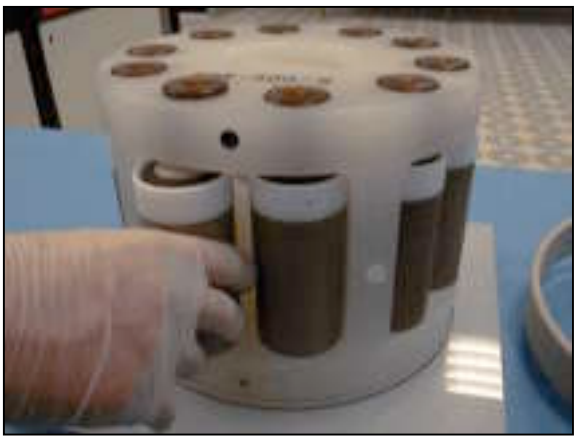
Place the rotor complete with vessels on the work station, located under a fume hood.
Carefully loosen the screw in the upper part of the rotor body, by using the tension wrench.
Open vessels very slowly, since a certain pressure may still be present (depending on sample type) even if temperature is as low as room temperature.
Open the vessel opposite to your position, as shown by the next picture.

Get a good control of the opening step rotating the torque wrench with wide angles.

Remove the external protection ring (picture).



Take the vessels out of the rotor body (picture).



Carefully take up the vessel cover together with the TFM Teflon protection ring (picture).

Rinse the lower part of the TFM Teflon cover with distilled water, collecting the same inside the vessel (picture).

Slide the TFM Teflon vessel out of the HTC protection shield (picture).

Pour the solution in a laboratory volumetric flask, washing repeatedly the TFM Teflon vessel with distilled water (picture).

Repeat the same operation with all vessels in the rotor.
Your samples are now ready to be analysed.

1.8 MDR-1000/6S

The MDR-1000/6S high pressure rotor contains 6 vessels for acid digestion of routine and difficult samples at operating pressure up to 100bar (1.450psi).

1.8.1 MDR-1000/6S SETUP

The MDR-1000/6S has P/N 35301 and it is composed by the following items.

P/N	Quantity	Description
35320	1	Rotor body
34010	6	Safety spring
34019	6	Adapter plate
34043	6	TFM Teflon cover
34056	6	Indicator ring
34040	6	TFM Teflon vessel
34048	6	Safety shield

1.8.2 HOW TO OPERATE WITH THE MDR-1000/6S ROTOR

1.8.3 SAMPLE WEIGHING, REAGENTS ADDITION AND VESSEL CAPPING

Place a TFM Teflon vessel directly on the balance plate.
The weight of the TFM Teflon vessel is about 120g, therefore most analytical balances may be used for the pourpose (picture).

Tare the vessel and directly weigh the sample by placing it inside the vessel (picture).

Try to reduce as much as possible sample transfer, to avoid any contamination.
Introduce the TFM Teflon vessel into the HTC protection shield.

WARNING
The wall surfaces of the HTC protection shield as well as the outer wall of the TFM Teflon vessel must be dry and clean.
Damages may occour otherwise!

Add suitable reagents to the sample.
When part of the sample is left on the inner wall of the TFM Teflon vessel, try to wet it by adding acids drop by drop (picture).

Gently swirl the solution, to homogenise the sample with the acid.
Place the TFM Teflon cover on the TFM Teflon vessel.
Push it by end and check that the cover fits well onto the vessel (picture).

Place now the HTC adapter plate on the TFM Teflon cover, with the flat part of it at facing downwards, to have the space for the HTC safety spring on the top side (picture).

Locate the HTC safety spring onto the HTC adapter plate (picture).

Place the TFM Teflon indicator ring on the cover and push it down completely, until it fits the groove of the HTC safety shield, as shown by the next picture.

Position the polypropylene rotor body on the work station (picture).

Introduce the vessel vertically into one of the numbered niches carved along the periphery of the 6-position polypropylene rotor body.
Notice the position of the venting hole located in the TFM Teflon indicator ring.
The hole must face outside, so that in case of venting no hot acid vapors can damage the polypropylene rotor body (picture).

It may be useful now to fix the vessel inside its location, without a complete closing.
Take the torque wrench and remove the plastic adaptor (picture).

Use the adaptor to fix the vessels, as shown in the next picture.

Repeat the same operations for the remaining nine vessels; then put the external protection ring around the rotor body (picture).

Take the torque wrench and set the indicator to the “CLOSE” position, as shown in the next picture.

Tighten the HTC screw in the upper part of the rotor body using the torque wrench.
Make long rotations (refer to the two next pictures) instead of short ones and do proceed slowly until you hear a clicking sound informing that the vessel is properly closed inside its niche.

WARNING
Stop turning the torque wrench when the click is heard.

In fact the torque wrench may overload the HTC safety spring if further rotation is applied.

Make slow 90° rotations with the torque wrench, to get a better control of the correct closing of the vessel (click sound).

The MDR-1000/6S rotor is now ready to be introduced inside the ETHOS 900/1600; open the door of the microwave unit by pressing down the door handle (picture).

Slide-in the MDR-1000/6S rotor inside the microwave cavity of the ETHOS 900/1600 (picture).

Turn the rotor until it matches the drive adaptor located at the center of the microwave cavity base (picture).

Close the door of the ETHOS 900/1600 (picture).

Switch on the ETHOS 900/1600 (picture) and start a suitable microwave acid digestion program.

1.8.4 COOLING DOWN AND UNCAPPING THE VESSEL

Once the digestion program is completed, high temperature and pressure are reached inside the vessels, according with the sample preparation procedure applied.
It is therefore necessary to cool down the rotor before opening the vessels.
To facilitate this operating step Milestone has developed a cooling system able to reduce the cooling time to about 10 minutes.
Introduce the rotor body complete with vessels and protection ring inside the cooling system (picture above).

Put the cover back on the cooling system itself (picture).

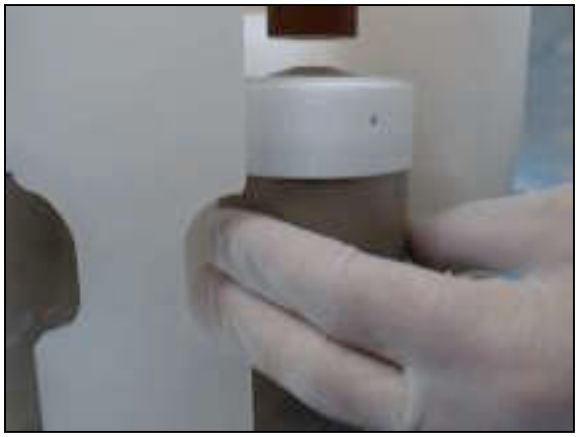
Turn on the water supply and let the water flow in for approximately 10-15 minutes (picture).

Turn off the water supply and move the rotor body from the cooling system to the work station, preferably located under a fume hood.
Dry with the utmost care rotor and vessels prior uncapping.
Take the torque wrench and set the indicator to the “OPEN” position, as shown in the next picture.

Place the rotor complete with vessels on the work station, located under a fume hood.
Carefully loosen the screw in the upper part of the rotor body, by using the tension wrench.
Open vessels very slowly, since a certain pressure may still be present (depending on sample type) even if temperature is as low as room temperature.
Open the vessel opposite to your position, as shown by the next two pictures.
Get a good control of the opening step rotating the torque wrench with wide angles.

Remove the external protection (picture).

Take the vessels out of the rotor body (picture).



Carefully take up the vessel cover together with the TFM Teflon protection ring (picture).

Rinse the lower part of the TFM Teflon cover with distilled water, collecting the same inside the vessel (picture).

Slide the TFM Teflon vessel out of the HTC protection shield (picture).

Pour the solution in a laboratory volumetric flask, washing repeatedly the TFM Teflon vessel with distilled water (picture).

Repeat the same operation with all vessels in the rotor.
Your samples are now ready to be analysed.

1.9 MDR ROTORS MAINTENANCE

As general rules:

- do not use any organic solvent to clean or dry any part of the rotor, with the exception of the TFM Teflon covers and of the TFM or PFA Teflon vessels.
- Dry every component including the vessels thoroughly with paper towel or cloth before placing the rotor in the microwave unit.
  Any moisture or acid trace would heat up in the microwave field and it could possibly damage the components.
  This is especially true if any trace of water or acid is left between the internal wall of the safety shields and the external walls of the vessels.
- Do not use ice to cool down the rotor after microwave heating, as the thermal shock could damage the safety shield.
- To wash all kind of vessels it is advisable to make use of one of the following procedures

1.9.1 RAPID CLEANING PROCEDURE

1. Add 5mL HNO_3 in each vessel;
2. close the vessels inside the rotor as for a normal digestion;
3. start the following microwave program (6 vessels): 10 minutes @ 500Watt or (10 vessels): 10 minutes @ 650Watt;
4. cool down and open the vessels;
5. rinse the vessels with bidistilled water.

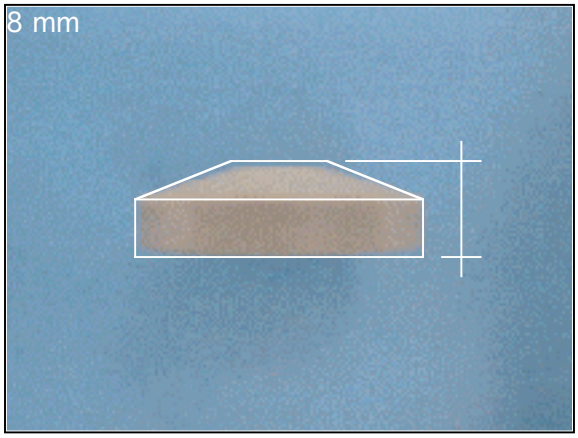
1.9.2 "US EPA" RECOMMANDED CLEANING PROCEDURE

The cleaning procedure briefly described here is part of the US EPA approved method 3052 for the microwave acid digestion of environmental samples.

1. Store TFM and PFA Teflon parts in 1:1 subboiled HCl overnight at 80-120°C;
2. rinse with bidistilled water;
3. Store in 1:1 subboiled HNO_3 overnight at 80-125°C;
4. rinse with bidistilled water;
5. store in bidistilled water;
6. allow parts to dry in a particle and fume-free environment.

1.9.3 SAFETY SPRING

After long time digestions and long term use the HTC-made springs are flattened and lowered.
The lifetime of the spring may vary depending on the working conditions (pressure, temperature) of the rotor, but it can be estimated in approximately 100+ digestions.
A new spring is 8,0mm thick (picture).



If the lowering measures less than 0,7mm, replacement is necessary.
A special propylene tool (P/N 40053) able to check the spring thickness is available from Milestone.
As shown by the picture below, the slot measures 7,3mm; if the spring can be introduced without forcing inside the slot, then the spring has to be replaced.

WARNING
It is mandatory to replace springs with thickness lower than 7,3mm.
Their use may cause severe damages to other components of the rotor.

1.9.4 HTC ADAPTER PLATE, TFM TEFLON COVER, TFM TEFLON INDICATOR RINGS AND TFM OR PFA TEFLON VESSEL

The HTC adapter plates, the TFM Teflon covers, the TFM Teflon indicator rings and the TFM or PFA Teflon vessels (shown in a picture below) do not practically undergo any deformation if properly used.
They have to be replaced only in case of damage or deformation.
A visual inspection of the above items has to be done before every digestion.

1.9.5 PRESSURE SCREW

The pressure screw (picture) in the rotor may slightly lose smoothness.
Threads can be re-made by a Service Technician using a proper stainless steel tool.

1.9.6 SAFETY SHIELDS

After long time operation the safety shields may show some swelling due to the high heating conditions.
Lifetime is estimated to be hundreds of digestions.
Check for visible deformation at the bottom of the safety shields (approximately 2cm from the base at the level of the solution).
Putting two shields aside the deformation may be evidenced (picture) and replacement is necessary.

If HNO_3 is often used to digest samples, than the upper part of the safety shield may assume an orange-like color, due to a chemical attack of the HTC surface by the HNO_3.
This is normally not a problem, unless the mechanical properties of HTC are compromised.

1.0 SOFTWARE MODEL LEVEL 6.17 FOR ETHOS 900

1.1 HOW TO CREATE AND STORE A NEW PROGRAM

NOTE
In order to make understanding of the following chapter clearer and easier, we have opted for the representation only of the upper window shown by the control terminal display.

The following example will be described.

PROGRAM 1					
Step	Time (hh:mm:ss)	Power (Watt)	Pressure (bar)	Int. Temp. (°C)	Ext. Temp (°C)
1	00:05:00	250	3	150	70
2	00:05:00	400	6	180	80
3	00:04:00	500	10	210	80
Ventilation	00:03:00				

Switch on the main switch of the ETHOS 900, located on the front panel.
The control terminal "240" display will alternatively show:

*****	MLS GmbH MultiQUANT	*****

*****	MLS GmbH SW V 6.17 (M 9_1d77)	*****

After a few seconds the display will show:

F1=WORK	F2=EDIT
F3=READIN	F4=READOUT

Press "F2" key, the display will show:

EDIT PROGRAM	1
(1-20)	ENT=OK

Press "ENT" to confirm; the display will show:

TIME	hh:mm:ss	1/1 ENT=OK

Introduce the desired time of the 1st step (5 minutes in our case) by pressing "0", "0", "0" and "5" on the keyboard.
The display will show:

TIME	00:05:ss	1/1 ENT=OK

Press "ENT" to confirm; the display will show:

POWER (-> 900)	0 W	1/1 ENT=OK

It is now possible to enter the microwave power level of the 1st step (250Watt in our case).
Press "2" and "5" on the keyboard and the display will show:

POWER 1	250 W	1/1

(-> 900)		ENT=OK

Press “ENT” to confirm; the display will now request the values for pressure, internal temperature and external temperature.

WARNING
The values of pressure, internal temperature and external temperature shall be set only when the ETHOS 900 is supplied with either one of the following control devices: APC-55 (Automatic Pressure Control), ATC-300 (Automatic Temperature Control) and IRTC-500 (Infra Red Temperature Control).
If these devices are not installed in the ETHOS 900, simply press “ENT” three times to skip these steps.

The display will show:

PRESSURE (-> 100)	_ B	1/1 ENT=OK

Press “3” on the keyboard and the display will show:

PRESSURE (-> 100)	3 B	1/1 ENT=OK

Press “ENT” to confirm the value; the following message will appear:

TEMP 1 (-> 400)	_ °C	1/1 ENT=OK

Press “1”, “5” and “0” (150°C) on the keyboard and the display will show:

TEMP 1 (-> 400)	150 °C	1/1 ENT=OK

Press “ENT” to confirm the value; the following message will appear:

TEMP 2 (-> 500)	_ °C	1/1 ENT=OK

Press “7” and “0” (70°C) on the keyboard and the display will show:

TEMP 2 (-> 500)	70 °C	1/1 ENT=OK

Press “ENT” to confirm the value; the display will now show the 2nd step of the program 1.

TIME	hh:mm:ss	2/1 ENT=OK

Introduce the desired time of the 2nd step (5 minutes in our case) by pressing “0”, “0”, “0” and “5” on the keyboard. The display will show:

TIME	00:05:ss	2/1 ENT=OK

Press “ENT” to confirm; the display will show:

POWER (-> 900)	0 W	2/1 ENT=OK

It is now possible to enter the microwave power level of the 2nd step (400Watt in our case).
Press “4” and “0” on the keyboard and the display will show:

POWER 1 (-> 900)	400 W	2/1 ENT=OK

Press “ENT” to confirm; the display will show:

PRESSURE (-> 100)	_ B	2/1 ENT=OK

Press “6” on the keyboard and the display will show:

PRESSURE (-> 100)	6 B	2/1 ENT=OK

Press “ENT” to confirm the value; the following message will appear:

TEMP 1 (-> 400)	_ °C	2/1 ENT=OK

Press “1”, “8” and “0” (150°C) on the keyboard and the display will show:

TEMP 1 (-> 400)	180 °C	2/1 ENT=OK

Press “ENT” to confirm the value; the following message will appear:

TEMP 2 (-> 500)	_ °C	2/1 ENT=OK

Press “8” and “0” (80°C) on the keyboard and the display will show:

TEMP 2 (-> 500)	80 °C	2/1 ENT=OK

Press “ENT” to confirm the value; the display will now show the 3rd step of the program 1.

TIME	hh:mm:ss	3/1 ENT=OK

Introduce the desired time of the 3rd step (4 minutes in our case) by pressing “0”, “0”, “0” and “4” on the keyboard.
The display will show:

TIME	00:04:ss	3/1 ENT=OK

Press “ENT” to confirm; the display will show:

POWER (-> 900)	0 W	3/1 ENT=OK

It is now possible to enter the microwave power level of the 3rd step (500Watt in our case).
Press “5” and “0” on the keyboard and the display will show:

POWER 1 (-> 900)	500 W	3/1 ENT=OK

Press “ENT” to confirm; the display will show:

PRESSURE (-> 100)	_ B	3/1 ENT=OK

Press “6” on the keyboard and the display will show:

PRESSURE (-> 100)	10 B	3/1 ENT=OK

Press “ENT” to confirm the value; the following message will appear:

TEMP 1 (-> 400)	_ °C	3/1 ENT=OK

Press “2”, “1” and “0” (210°C) on the keyboard and the display will show:

TEMP 1 (-> 400)	210 °C	3/1 ENT=OK

Press “ENT” to confirm the value; the following message will appear:

TEMP 2 (-> 500)	_ °C	3/1 ENT=OK

Press “8” and “0” (80°C) on the keyboard and the display will show:

TEMP 2 (-> 500)	80 °C	3/1 ENT=OK

Press “ENT” to confirm the value; the display will now show the 4th step of the program 1.

TIME	hh:mm:ss	4/1 ENT=OK

Our example program requires only 3 steps.
To skip step 4 through step 10 simply press “ENT” on the keyboard.
The display will show:

ROTOR CTRL=OFF MODE 1=ON/OFF	1 ENT=OK

Standard setting is ROTOR CTRL=OFF.
This function may be set ON when the optional IRTC-500 Infra Red Temperature Control device is installed in the ETHOS 900.
Press the key “MODE 1” to switch the function to ON; then confirm by pressing “ENT” on the keyboard.
The display will now show:

TIME AT TEMPERATURE 1=OFF MODE 1=ON/OFF	1 ENT=OK

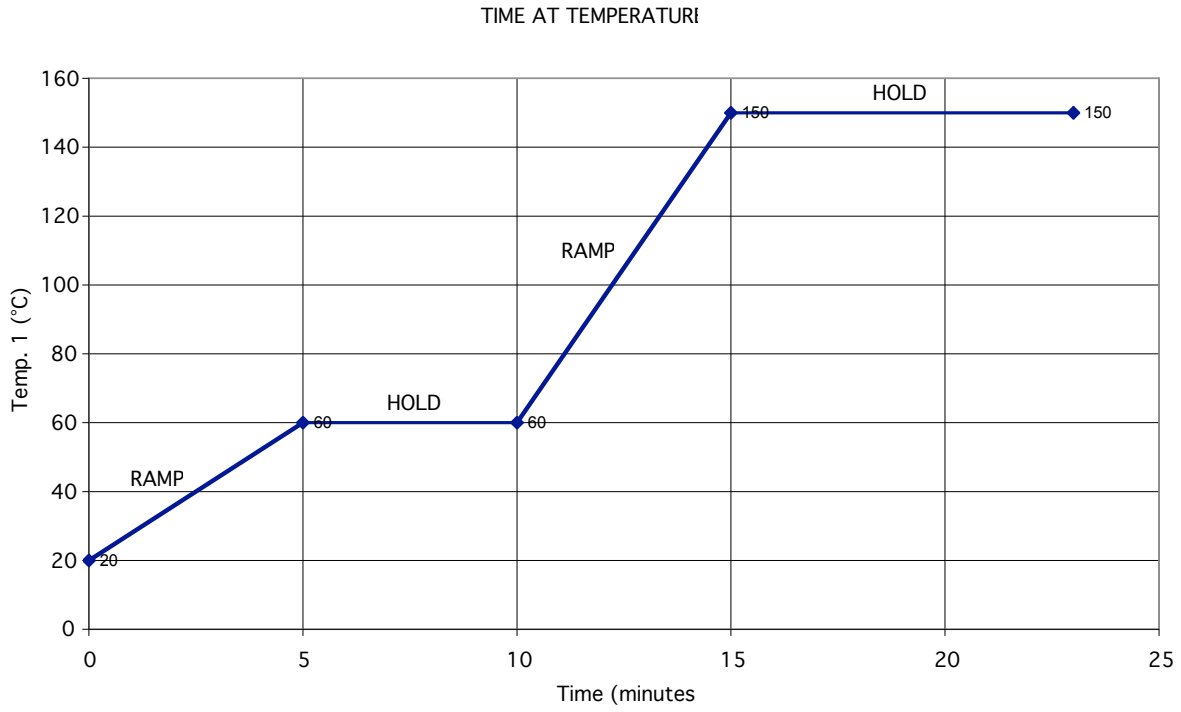
Standard setting is TIME AT TEMPERATURE=OFF.

The “TIME AT TEMPERATURE” function may be used when the ETHOS 900 is supplied with an optional ATC-300 Automatic Temperature Control device.
It allows to hold preset temperature values for preset times.
Press the key “ENT” if an ATC-300 is not installed in the ETHOS 900 or if the “TIME AT TEMPERATURE” function is not required.
By pressing the key “MODE 1”, the “TIME AT TEMPERATURE” function is activated.
In the latter case, the microwave program will be composed by:

- ramping steps: step 1, 3, 5, 7 and 9;
- holding steps: step 2, 4, 6, 8 and 10.

The diagram below better explains the “TIME AT TEMPERATURE” function.

Step	Time (hh:mm:ss)	Power (Watt)	Pressure (bar)	Temp. 1 (°C)	Temp. 2 (°C)
1	00:05:00	500	0	60	0
2	00:05:00	500	0	60	0
3	00:05:00	800	0	150	0
4	00:08:00	800	0	150	0
5	00:00:00	0	0	0	0
6	00:00:00	0	0	0	0
7	00:00:00	0	0	0	0
8	00:00:00	0	0	0	0
9	00:00:00	0	0	0	0
10	00:00:00	0	0	0	0



If, during the 1st step, the temperature will reach 60°C, then the program will skip the remaining time of the 1st step to proceed directly with the 2nd step (time at temperature, 5 minutes at 60°C).
If, during the 3rd step, the temperature will reach 150°C, then the program will skip the remaining time of the 3rd step to proceed directly with the 4th step (time at temperature, 8 minutes at 150°C).
Press “ENT” on the keyboard and the display will show:

TWIST=ON	1
MODE 1=ON/OFF	ENT=OK

The “TWIST” function allows an alternate 360°C rotation of the carousel or of the rotor.
This is required when the ETHOS 900 comes with an APC-55 Automatic Pressure Control or with an ATC-300 Automatic Temperature Control device, to avoid bending of the Teflon connecting tubing between the reference vessel and the pressure transducer (APC-55) and to avoid the bending of the temperature sensor (ATC-300).
Press the key “MODE 1” to activate (ON) or deactivate (OFF) the “TWIST” function.
Press “ENT” on the keyboard and the display will show:

VENT	hh:mm:ss	1 ENT=OK

The ventilation step is very important and should not be omitted.
It serves to cool down the vessels inside the microwave cavity before removing the rotor for further water cooling.
It is therefore an important safety issue.
In the above mentioned example the ventilation step lasts 3 minutes; therefore digit 0, 0, 0 and 3.
The display will show:

VENT	00:03:ss	1 ENT=OK

Press “ENT” on the keyboard to confirm and the display will show:

PROGRAM 1 IS READY

And then:

F1=WORK	F2=EDIT
F3=READIN	F4=READOUT

End of program setting.

1.2 HOW TO VISUALIZE A STORED PROGRAM

Switch on the ETHOS 900 unit; the display will show:

F1=WORK	F2=EDIT
F3=READIN	F4=READOUT

Press the “F2” key; the display will show:

EDIT PROGRAM	*1
(1-20)	ENT=OK

The asterisk (*) indicates that the program number 1 has been previously stored.
Press the key “ENT” and the display will show:

PROGRAM 1 EXISTS	
DEL=NEW	ENT=OK

Press the key “ENT” to visualize in sequence the previously entered program and parameters.
To end the visualization, press the “STOP” key and the display will show:

EDIT PROGRAM 1
ABORTED!

And then, after pressing the “STOP” key, the display will show:

F1=WORK	F2=EDIT
F3=READIN	F4=READOUT

1.3 HOW TO DELETE A PROGRAM

F1=WORK	F2=EDIT
F3=READIN	F4=READOUT

Press the “F2” key and the display will show:

EDIT PROGRAM	*1
(1-20)	ENT=OK

Press the “ENT” key and the display will show:

	PROGRAM 1 EXISTS	
DEL=NEW		ENT=OK

Press the “DEL” key and the display will show:

TIME	Hh:mm:ss	1/1
		ENT=OK

The previous program has been deleted and a new program can be stored.
Cancellation of a program can be verified as no asterisk (*) will appear before the program number.
Press the “STOP” key twice and the display will show:

F1=WORK	F2=EDIT
F3=READIN	F4=READOUT

1.4 HOW TO DELETE ALL PROGRAMS

F1=WORK	F2=EDIT
F3=READIN	F4=READOUT

Press the “F2” key and the display will show:

EDIT PROGRAM	*n
(1-20)	ENT=OK

Press the “0” key and the display will show:

EDIT PROGRAM	_
(1-20)	ENT=OK

The symbol “_” will blink.
Press the “ENT” key and the display will show:

KILL ALL PROGRAMS	
SURE?	DEL=YES

When the “DEL” key is pressed, all the programs in memory will be cancelled; if the “STOP” key is pressed twice, then the display will return to the main menu.

1.5 HOW TO VERIFY DELETION OF A PROGRAM

F1=WORK	F2=EDIT
F3=READIN	F4=READOUT

Press the “F1” key and the display will show:

WORK PROGRAM	1
(1-20)	ENT=OK

Press the “ENT” key and the display will show:

PROGRAM 1
NOT FOUND

1.6 ARROWS FUNCTIONS

When the display shows:

WORK PROGRAM	*1
(1-20)	ENT=OK

The arrow keys allow selection of the work program sequence (for instance upwards from 1 to 2, 3 etc. with -> and downwards from 3 to 2, 1 etc. with <-).
When the display shows:

EDIT PROGRAM	1
(1-20)	ENT=OK

The arrow keys allow to select the program to be created.
Note that the arrow keys allow the selection of the stored programs only in both work and edit mode.

1.7 HOW TO MODIFY A PREVIOUSLY STORED PROGRAM

When the display shows:

	PROGRAM 1 EXISTS	
DEL=NEW		ENT=OK

Press the “ENT” key and the display will show:

TIME	00:05:00	1/1
		ENT=OK

Using the arrow keys as well as the number keys, it is possible to modify microwave emission time, power, parameters as P, T1 and T2 in each of the ten steps included in the program.
As soon as the parameter has been modified, press the “STOP” key to come back to the main menu.

1.8 PARAMETERS CHOICE

1.8.1 MICROWAVE POWER

The desired microwave power can be set in 10Watt increments up to a maximum of 900Watt.
For any value, for instance 300Watt, it is sufficient to introduce 30 as the first 0 is already fixed.

1.8.2 PRESSURE

The APC-55E Automatic Pressure Control is required.
A pressure up to 55bar can be set (when operating with a MDR-1000/6S rotor).
At the set pressure level the microwave emission will be controlled.

1.8.3 TEMPERATURE 1

The ATC-300 Automatic Temperature Control is required.
A temperature up to 240°C can be set, starting from which the microwave emission will be controlled.

1.8.4 TEMPERATURE 2

The IRTC-500 Infrared Temperature Control is required.
An external temperature up to 120°C can be set, starting from which the microwave emission will be controlled.

1.8.5 ROTOR CONTROL

Rotor control ON/OFF.
When operating with an IRTC-500, if rotor control is set ON, then the infrared sensor will read the highest value out of a complete turn of the rotor.
This value will be used for controlling the microwave output emission.

1.8.6 TWIST

The “twist” function must be activated in case of pressure and internal temperature control in the reaction vessels.
A 360° alternate rotation of the MDRs is activated, to avoid bending of the sensor connections.
In case the “twist” function is off, the unit presents no alternate rotation and before program running the following message will appear “TWIST NOT READY” and “POWER IS OFF”.

1.8.7 VENTILATION

Insert the ventilation time to cool down the vessels prior water cooling.

1.9 HOW TO USE A PREVIOUSLY STORED PROGRAM

Switch on the ETHOS 900 and the display will show alternatively the two following messages:

*****	MLS GmbH MultiQUANT	*****

*****	MLS GmbH SW V 6.01 (M10 1d77)	*****

After a few seconds the display will show:

F1=WORK	F2=EDIT
F3=READIN	F4=READOUT

Press the “F1” key to activate the “WORK” function; the display shows:

WORK PROGRAM	*1
(1-20)	ENT=OK

Select the number corresponding to the program to be used (for instance, 1).
Confirm the choice of program number 1 by pressing the “ENT” key and the display shows:

START=RUNNING PRG		1
P=0	T1=0	T2=0

Note that when the APC-55E, the ATC-300E or the IRTC-500 options are installed in the ETHOS 900, the actual values of pressure, internal temperature and external temperature will be shown.
Press the “START” key to start the program number 1; the display shows:

0W	00:00:05 CHECK AND START	P1

During the brief pause that follows, the magnetron will be warmed-up; after 5 seconds the program is started.
During running of the program the display shows, step by step, the microwave power (in Watt), the time remaining (in hours, minutes and seconds) and the actual values of pressure, internal temperature and external temperature.
However the following circumstances may occur:

>DOOR NOT CLOSED<

The door of the ETHOS 900 has not been properly closed; the microwave program is hold until the door is closed.

>TWIST NOT READY<

The “twist” function is not properly working; this may cause damages to the pressure and temperature sensors.
The microwave program is interrupted.
The microwave program can be stopped at anytime by pressing the “STOP” key or by lowering the door handle.
Pressing the “STOP” key, the display shows:

USER BREAK

Only if ventilation time was preset, the program will automatically proceed with the ventilation time.
After the ventilation time, the display shows:

	PROGRAM 1 IS READY POWER IS OFF

On the contrary, if no ventilation time was preset, the display shows immediately the above message.
To restart the program it is necessary to press the “START” key twice.
In case the microwave program is stopped by lowering the ETHOS 900 door handle, the four built-in switches are activated and the display shows:

	DOOR NOT CLOSED

Followed by:

	CONTINUE=CLOSE DOOR	
P=0	T1=0	T2=0

After closing the door, the program starts again exactly where it had been interrupted, whereas pressing “STOP” the display shows:

START=RUNNING PRG		1
P=0	T1=0	T2=0

Press “START” to make the program start from the beginning.

1.10 SETUP

Anytime, whatever the message on the display (obviously with the ETHOS 900 in stand-by position), it is possible to run the “SETUP” program, by pressing the “F6” key on the control terminal.
The display shows:

F1=SYSTEM	F2=VALUES
F3=BALANCE	F4=PC

Press the “F1” key to visualize the “SYSTEM” sub-menu; the display shows:

DATE	30.03.1998
	ENT=OK

Adjust the date, if necessary, by pressing the appropriate numbers on the keyboard; then confirm by pressing the “ENT” key.
The display shows:

TIME	11:55:00
	ENT=OK

Adjust the time, if necessary, by pressing the appropriate numbers on the keyboard; then confirm by pressing the “ENT” key.
The display shows:

BALANCE TYPE	2
(1,2,3)	ENT=OK

Adjust the type of balance to be interface by pressing 1, 2 or 3.
Default value is 2; confirm by pressing the “ENT” key and the display shows:

PULSE WIDTH	50/10s
(1-50)	ENT=OK

The ETHOS 900 operate with “unpulsed” emission of microwave at 250Watt.
When other power levels are selected, then a “pulsed” mode is used.
The standard setting for pulse width is 10/10s, equal to 1 second.
Microwave power is therefore regulated with alternate ON/OFF microwave emission, with duty cycles of 1 seconds, as for the following examples:

- 450Watt is equal to 0,5 seconds of magnetron ON and 0,5 seconds of magnetron OFF;
- 90Watt is equal to 0,1 seconds of magnetron ON and 0,9 seconds of magnetron OFF.

If the operator select a pulse width of 50/10s, equal to 5 seconds, then the following table will apply:

- 450Watt is equal to 2,5 seconds of magnetron ON and 2,5 seconds of magnetron OFF;
- 90Watt is equal to 0,5 seconds of magnetron ON and 4,5 seconds of magnetron OFF.

Confirm the choice of pulse width by pressing the “ENT” key and the display shows:

TWIST CHECK: ON	
MODE1=OFF	ENT=OK

This function enables the electronic check of the twist function.
Default mode is “ON”.
Press the key “MODE1” to disable the function; it is however recommended to leave it “ON” when the APC and/or the ATC sensors are installed.
Press the key “ENT” to confirm and the display shows:

GAS SENSOR	OFF

MODE1=SELECT (0)	ENT=OK

If a gas sensor is installed in the ETHOS 900, it can be activated with this section of the software.
Also the gas sensor sensitivity can be set.
There are four sensitivity steps: HIGH, MEDIUM, LOW and OFF.
It is possible to select one of these possibilities with the “MODE1” key.
When the gas sensor is activated, during a microwave program run microwave are turned OFF if the preset level is reached.
A warning message appears on the display.
When the gas concentration decreases below the preset level, the microwave program is continued and the warning message disappear.
Select HIGH, MEDIUM, LOW or OFF and confirm the choice by pressing “ENT” key.
The display shows:

DATE	30.03.98 ENT=OK

Press the “STOP” key to leave the “SYSTEM” sub-menu and the display shows:

F1=SYSTEM	F2=VALUES
F3=BALANCE	F4=PC

Press the “F2” key to visualize the “VALUES” sub-menu; the display shows the software model level, as well as the actual values of pressure, internal temperature and external temperature.

	SW_V: 6.17 (M 9_0d77)	
P=0	T1=0	T2=0

Press the “STOP” key to leave the “VALUES” sub-menu and the display shows:

F1=SYSTEM	F2=VALUES
F3=BALANCE	F4=PC

Press the “F3” key to visualize the “BALANCE” sub-menu; the display shows direct readout of the balance, when this is connected.

WEIGHT=	0.2308g
0=ZERO	ENT=PRINT

Press the “0” key to tare the balance or press the “ENT” key to printout the balance readout.
By pressing the “STOP” key the display turns back to the main menu:

F1=SYSTEM	F2=VALUES
F3=BALANCE	F4=PC

Press “F4” to interface the ETHOS 900 to an external computer.
By an appropriate software the ETHOS 900 is fully operated by an external computer.

	COMPUTER ON LINE

Press the “STOP” key to return to the main menu.
By pressing the “F5” key from the main menu a printout of whatever the display shows is activated.

1.11 ERROR MESSAGES

In case the operator has any difficulty with the control terminal software, the basic rule is to press the “STOP” key to return to the main menu.
Switching ON/OFF the unit will cause resetting and going back to the main menu.
Work programs remain unchanged.

1.11.1 TWIST FUNCTION

This function has been introduced for higher safety of operation.
Basic setting is TWIST=ON.
If the operator is using an MDR rotor fitted with pressure and/or internal temperature control, the twist function must be ON, as otherwise the sensor tubing will be damaged.

```
TWIST not ready!
-POWER IS OFF-
```

1.11.2 ROTOR CONTROL

If the ETHOS 900 is fitted with an IRTC-500, it is important to activate the rotor control; therefore at the following step:

```
ROTOR-CTRL=ON1
MODE1-ON/OFF   ENT=OK
```

1.11.3 OVERHEATING

As an extra safety the ETHOS 900 magnetron are provided with overheating thermal safety switches.
In the remote event of hardware overheating, the following message appears:

```
>DOOR NOT CLOSED<
```

And then:

```
CONTINUE=CLOSE DOOR
ABORT=STOP
```

If this error message is not due to the ETHOS 900 door left open, then interrupt the microwave program.
After cooling, the system can be regularly operated again.

1.11.4 BATTERY

The ETHOS 900 CPU is provided with a Lithium battery to save the storing in memory of the working programs.
The lifetime of the memory buffer battery is approximately 3 years and therefore it should be replaced after this time period.
If the battery is empty, the software will give a user message and delete all programs.

```
BATTERY IS EMPTY
ALL PROGRAMS ARE KILLED
```

A double beep follows this message.
If you still want to work with a low battery, you have to enter the whole program after power on the ETHOS 900.

1.11.5 LONG TIME OPERATION PROGRAM

All reactions over 30 minutes and above 250Watt should only be carried out with the ATC-300 Automatic Temperature Control device installed and in use and only after contacting first the Milestone representative in your country for advise.
The next function has been introduced to protect the ETHOS 900 from overheating problems caused by long time programs (more than 1 hour).
If the program being used is longer than 1 hour, when pressing F1=WORK a three times warning message appears:

**	WARNING TOTAL PRG TIME > 1h	**

Press the “ENT” key and the display shows:

**	ARE YOU SURE TOTAL PRG TIME > 1h	**

Press the “ENT” key and the display shows:

**	ARE YOU REALLY SURE TOTAL PRG TIME > 1h	**

Press the “ENT” key and the display shows:

	START=RUNNING PRG ...	
P=0	T1=0	T2=0

Now the program is available to work.
Otherwise if you want to change the program, press the “STOP” key when the warning message appears, then press again the “STOP” key until the main menu is reached.

F1=WORK	F2=EDIT
F3=READIN	F4=READOUT

1.12 SOFTWARE INITIALIZATION

Every time either a CPU board or a EPROM is replaced, the system needs to be reinitialized.
Proceed as follows:

- start with ETHOS 900 in OFF conditions;
- press the “STOP” key on the control terminal;
- keeping pressed, switch ON the ETHOS 900;
- remain in this position until a buzzer sound is heard;
- enter a new working program as previously described.

1.13 MULTIQUANT MODE

The ETHOS 900, besides being a digestion system, can also operate as moisture/solids analyzer.
For determination of volatile elements the operator must go to the program part “READIN” by pressing the “F3” key from the main menu.
Refer to this section only when a balance and a printer are connected to the ETHOS 900.
The basic principle of the multiQUANT mode is that volatile/moisture in samples are determined by weighing the samples themselves before and after a drying process.
The difference between the initial weight (“read-in”) and the final weigh (“read-out”) is determined and statistically calculated.
Quitting the main menu and pressing “F3=READIN”, the operator gets into the multiQUANT menu.
Introduce the sample identification (maximum 9 letters) replacing the writing MLS GmbH word.

- ID NO.
  Use the arrow keys to go forward and backwards in the alphabet; use the asterisk (*) to switch from capital to small letters; use the 0... 9 keys to write numbers.
- NO. SAMPLES
  Indicates the number of samples to be weighed/tested.
- 1 TARA
  Store the tare of the 1st sample.
- 1 TARA+S
  The 1st sample is weighed and the value is stored in memory.
- 2 TARA
  Store the tare of the 2nd sample.
- 2 TARA+S
  The 2nd sample is weighed and the value is stored in memory, and so on.
- F4=READOUT
  Weighing of final weight and statistical calculation with print-out of reports.
  In case during weighing, both read-in and read-out, there are one or more errors (sample lost/burnt, defective vessel etc.), it is not necessary to repeat the complete sequence, but single values can be rejected with the “READOUT” step.
  An exclamation mark will appear and the value will remain out of the statistical calculation.
  The procedure is reversible.
  Before quitting with the “STOP” key, it is suggested to press the “F5” key to have printout of the modified values.

1.0 GUIDELINES FOR MICROWAVE ACID DIGESTION

1.1 INTRODUCTION TO THE REAGENTS MOST COMMONLY USED IN MICROWAVE DIGESTION

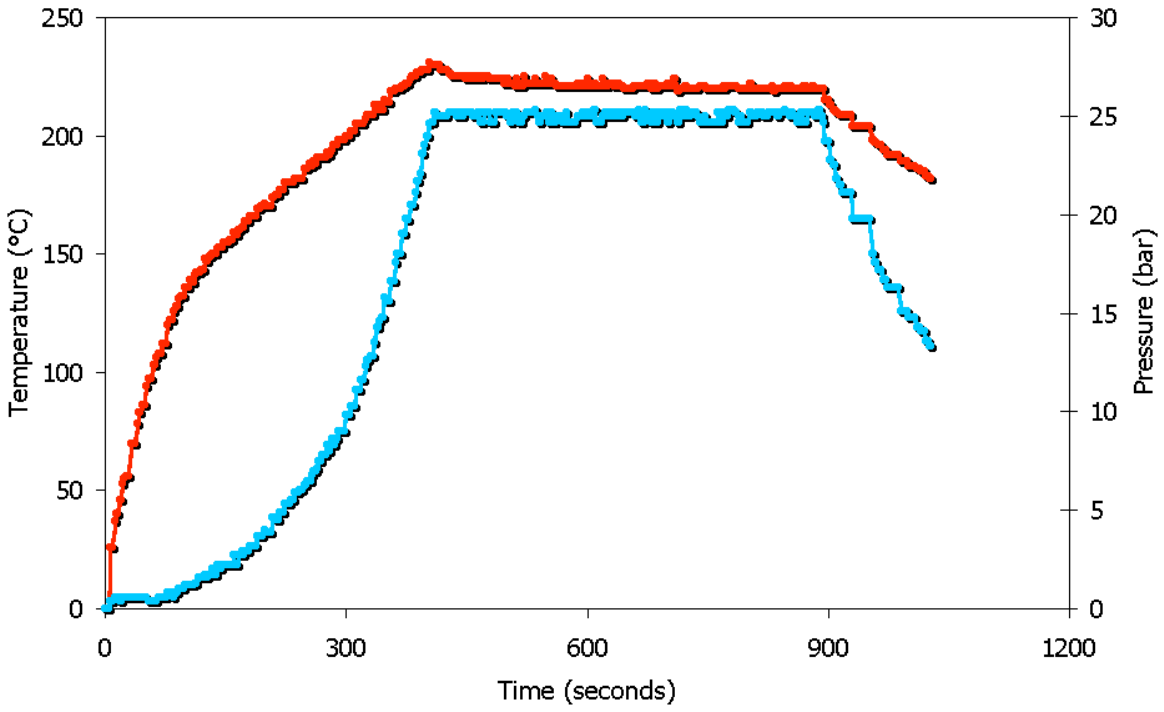
The acids used in microwave digestion may be classified in two main groups:

- nonoxidizing acids, such as hydrochloric acid, hydrofluoric acid, phosphoric acid, diluted sulfuric acid and diluted perchloric acid;
- oxidizing acids, such as nitric acid, hot concentrated perchloric acid, concentrated sulfuric acid and hydrogen peroxide.

1.2 NITRIC ACID

Nitric acid has the following properties:

- boiling point is 120°C at 65% concentration;
- poor oxidizing strength at concentrations less than 2M; oxidizing strength increases with concentration and reaction temperature;
- most common acid for oxidation of organic matrices with this typical reaction: $(CH_2)_X + 2HNO_3 \rightarrow CO_2(g) + 2NO + 2H_2O$;
- it dissolves most metals forming soluble nitrates, exceptions are Au and Pt (not oxidated) and Al, B, Cr, Ti and Zr (passivated);
- these metals require acid mixtures or diluted nitric acid;
- often mixed with H_2O_2, HCl and H_2SO_4;
- available in high purity for trace level analysis.



The above graphic represents the temperature and pressure profile of nitric acid up on microwave heating. Notice that pressure has been controlled at 25bar, resulting in a temperature of 225°C.

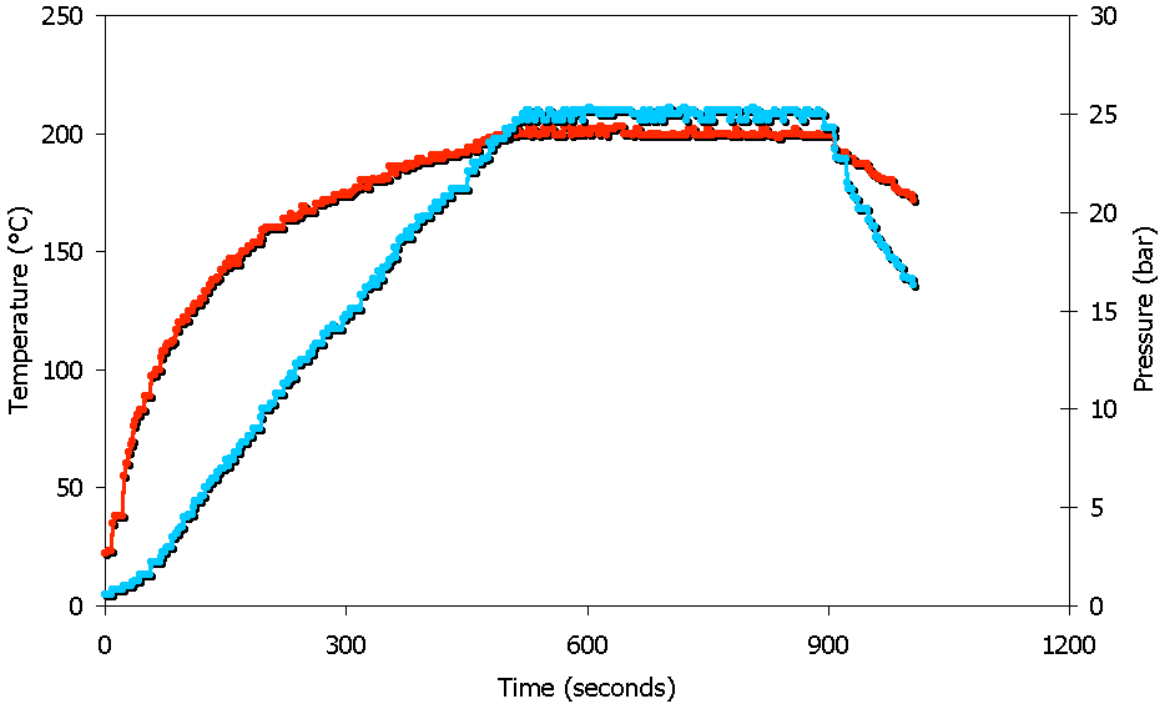
1.3 HYDROGEN PEROXIDE

Hydrogen peroxide is an oxidizing agent ($2H_2O_2 \rightarrow 2H_2O + O_2$); added to nitric acid it reduces the nitrous vapours and it accelerates the digestion of organic samples by raising the temperature.
A typical mixture ratio is HNO_3:H_2O_2= 4:1 (volume/volume).

1.4 HYDROCHLORIC ACID

Hydrochloric acid has the following properties:

- boiling point of azeotropic mixture with H_2O with 20,4% HCl is 110°C;
- available with 38% concentration;
- it dissolves salts of weak acids (carbonates, phosphates) and most metals are soluble with the exception of AgCl, HgCl and TiCl;
- excess of HCl improves the solubility of AgCl, converted into $AgCl_2^-$;
- strong complexing nature;
- widely used for iron-based alloys because of its ability to hold large amounts of chloro-complex in solution;
- other complexes formed are Ag (I), Au (II), Hg (II), Ga (III), Tl (III), Sn (IV), Fe (II) and Fe (III);
- it does not dissolve oxides of Al, Be, Cr, Ti, Zr, Sn and Sb; sulphates of Ba and Pb, group II fluorides, SiO_2, TiO_2 and ZrO_2.



The graphic above represents the temperature and pressure profile of hydrochloric acid up on microwave heating.
Notice that pressure has been controlled at 25bar, resulting in a temperature of 205°C.

1.5 HYDROFLUORIC ACID

Hydrochloric acid has the following properties:

1.5.1 ACID DIGESTION

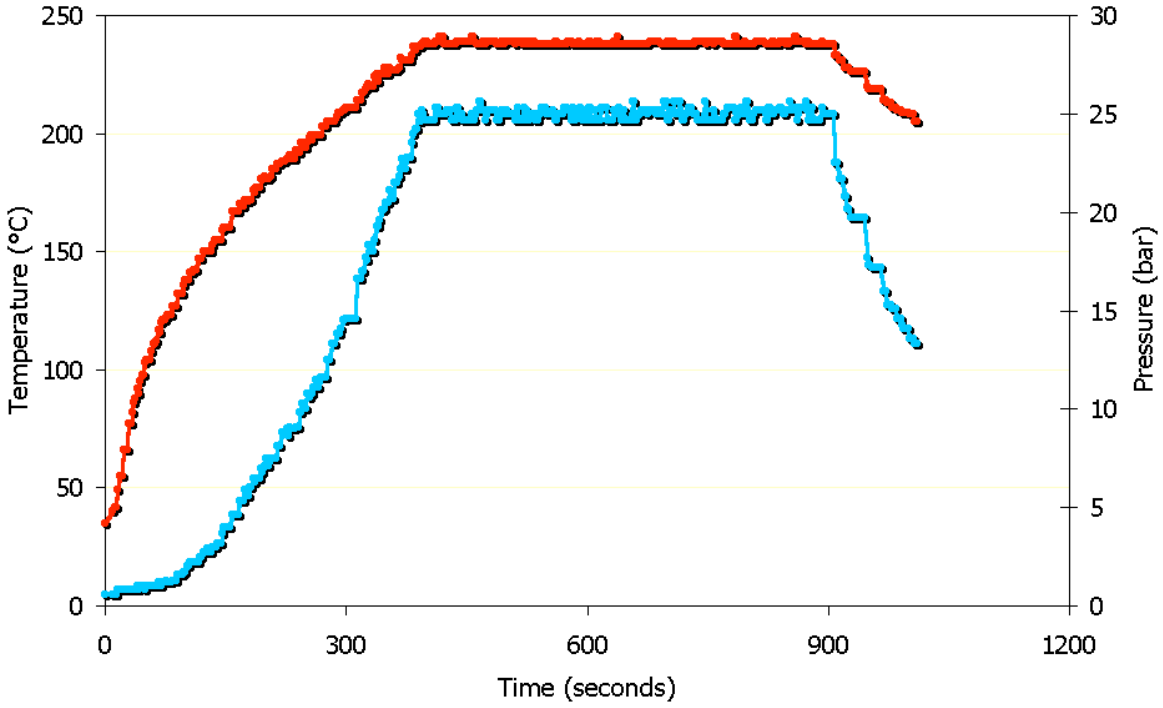
- Boiling point is 108°C at 40% concentration;
- nonoxidizing, strong complexing nature;
- used in digestion of minerals, ores, soils, rocks and even vegetables containing silicates;
- major use is the decomposition of silicates according with this reaction: $SiO_2 + 6HF \rightarrow H_2SiF_6 + 2H_2O$;
- often used in combination with HNO_3 or $HClO_4$.

1.5.2 EVAPORATION/CONCENTRATION

- Following dissolution, many analyses require removal of HF to prevent equipment damage or to resolubilize insoluble fluorides;
- many analytes such as As, B, Se, Sb, Hg and Cr may volatilize.

1.5.3 COMPLEXATION

- Alternative approach to remove HF from the solution, by addition of Boric acid;
- the following reactions take place: $H_3BO_3 + 3HF \rightarrow HBF_3(OH) + 2H_2O$ and $HBF_3(OH) + HF \rightarrow HBF_3 + H2O$;
- 10-50 times excess Boric acid enhances reaction rate.

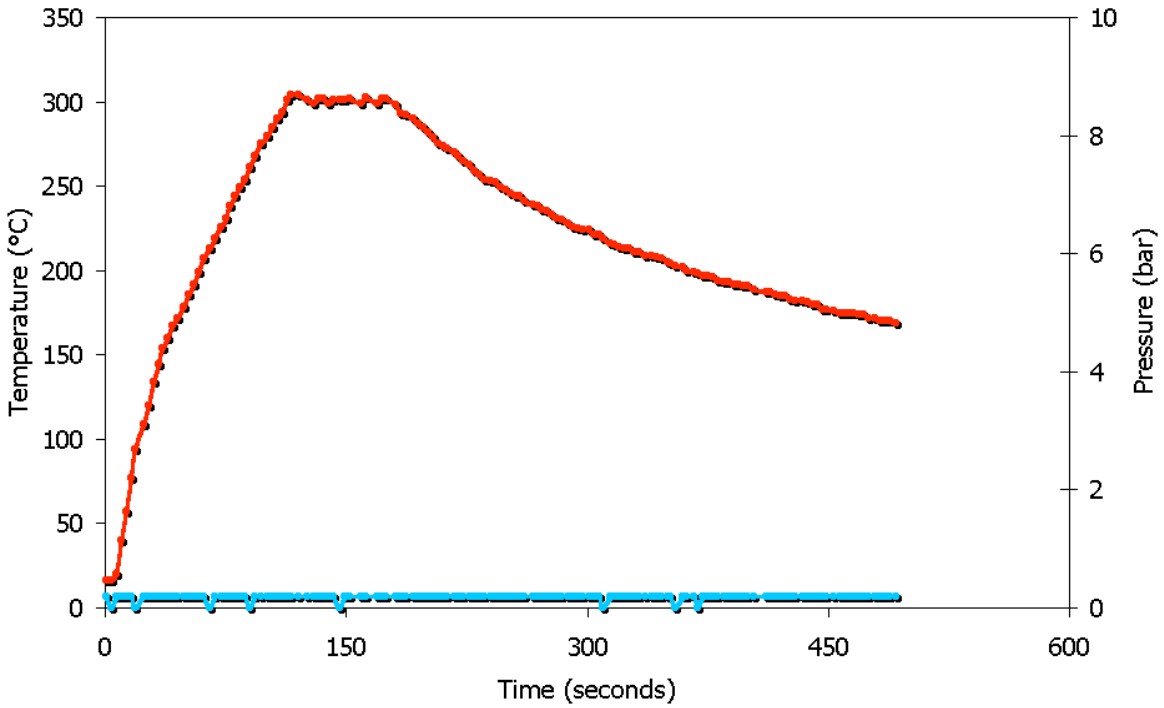


The above graphic represents the temperature and pressure profile of hydrofluoric acid up on microwave heating. Notice that pressure has been controlled at 25bar, resulting in a temperature of 240°C.

1.6 SULFURIC ACID

Sulfuric acid has the following properties:

- boiling point is 340°C at 98% concentration, exceeding the maximum working temperature of TFM Teflon vessels;
- careful reaction monitoring is required to prevent vessel damages;
- it destroys organics by dehydrating action;
- many sulfates are insoluble (Ba, Sr, Pb).



The above graphic represents the temperature and pressure profile of sulfuric acid up on microwave heating.
Notice that temperature has been controlled at 300°C (for 1 minute only), without any pressure increase.
It is clear that the use of concentrated sulfuric acid in the MDR rotors poses significant problems because of its high boiling point.
300°C are critical for TFM Teflon vessels and simply too high for PFA Teflon vessels (they melt down at such temperature).
It is therefore advisable to use sulfuric acid only with a strict temperature control.

1.7 PERCHLORIC ACID

Perchloric acid has the following properties:

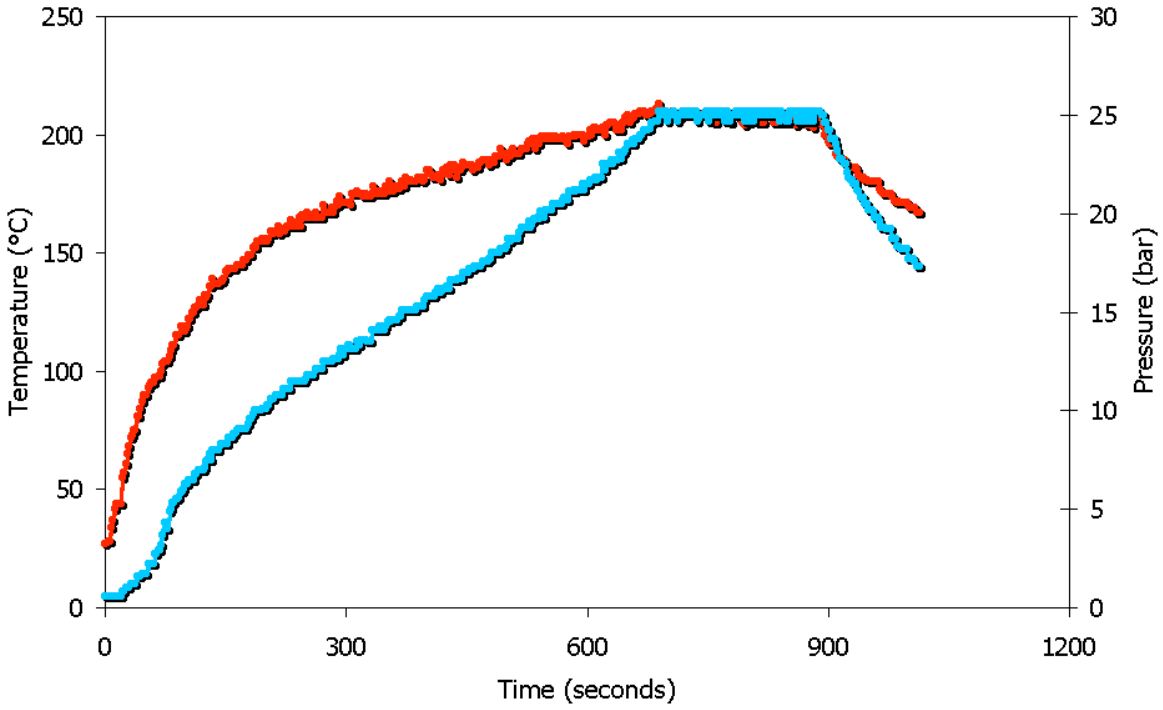
- boiling point is 203°C at 72% concentration;
- hot and concentrated is the strongest oxidizing acid;
- rapid, sometimes explosive, reaction with organic matrices;
- often mixed with nitric acid for a controllable digestion of organic matrices;
- all perchlorate are soluble with the exception of $KClO_4$;
- perchloric acid decomposes at 245°C in a closed microwave vessel, developing gaseous by-products and a tremendous excess pressure.

WARNING
Special care has to be taken when the use of perchloric acid is required.
Do not use at all perchloric acid with organic samples.
It may be possible to use perchloric acid with inorganic samples when the temperature does not exceed 200°C and when the amount of perchloric acid is below 20% (volume/volume) of the total solution volume.

1.8 AQUA REGIA

Aqua regia properties are the following:

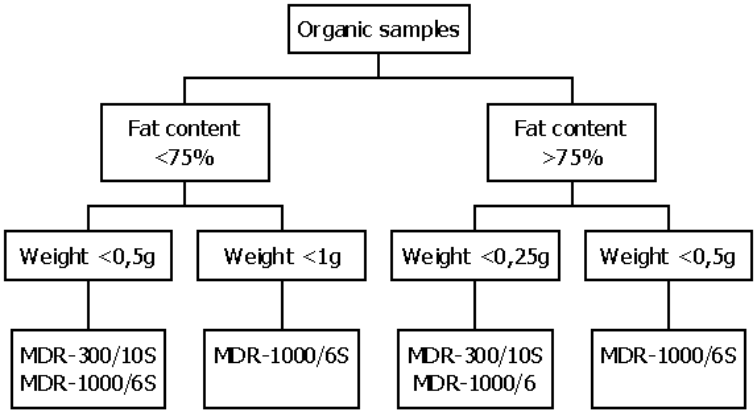
- made up by hydrochloric acid and nitric acid in a 3:1 (volume/volume) mixture;
- it produces NOCl (nitrosyl chloride), which decomposes in NO and Cl_2 up on heating;
- it dissolves precious metals;
- it must be freshly prepared and used immediately, otherwise it evolves chlorine gas overpressurizing and venting the vessel



The above graphic represents the temperature and pressure profile of aqua regia up on microwave heating. Notice that pressure has been controlled at 25bar, resulting in a temperature of 200°C.

2.0 MICROWAVE ACID DIGESTION OF ORGANIC SAMPLES

Organic samples group includes food, feed, tissues, botanicals, biological etc.
Nitric acid is the most common oxidizing agent used to digest organic samples, according to the following reaction: ORG + HNO_3 -> NO_X + CO_2 + H_2O.
Metals are converted into soluble nitrates, available for analysis.
When operating the ETHOS 900 with ATC-300 Automatic Temperature Control device, set the digestion temperature at 140°C or higher for samples rich in carbohydrates (wheat, sugar, etc.), at 150°C or higher for samples rich in proteins (serum, albumin etc.) and at 170°C or higher for fatty samples (cheese, butter, vegetable oil etc.).
Those temperature represent minimum target digestion temperature for the above matrices.
The sample amount directly influence the pressure generated inside the vessel, as sample is decomposed into CO_2 gas.
Therefore some limitation in regards to the sample amount may apply.
Follow the scheme below.



Note that the above sample amounts do refer to organic matter.
Therefore the moisture content of the sample should not be considered (for instance, if a sample contains about 80% of moisture, up to 5 gram could be digested).
When operating the ETHOS 900 without ATC-300 Automatic Temperature Control, follow the basic programs recommended by Milestone.
Microwave power and heating time strictly depend on the number of samples being prepared simultaneously.
To organic samples 4-8mL of nitric acid (depending on sample amount) should be added, as well as 1-2mL of hydrogen peroxide.

2.1 BASIC PROGRAM FOR 3 SAMPLES

Step	Time (minutes)	Power (Watt)
1	5	250
2	3	600
3	2	300

2.2 BASIC PROGRAM FOR 6 SAMPLES

Step	Time (minutes)	Power (Watt)
1	5	250
2	5	500
3	5	650

2.3 BASIC PROGRAM FOR 12 SAMPLES

Step	Time (minutes)	Power (Watt)
1	8	250
2	8	500
3	8	750

3.0 MICROWAVE ACID DIGESTION OF ENVIRONMENTAL SAMPLES

A number of microwave digestion methods for environmental samples have been approved by the US EPA (Environmental Protection Agency).

- US EPA 3015 SW-846 Update II
  Microwave assisted acid digestion of aqueous samples
- US EPA 3051 SW-846 Update II
  Microwave assisted acid digestion of sediments, slugs, soils and oils
- US EPA 3052 SW-846 Update III
  Microwave assisted acid digestion of siliceous and organically based matrices

A brief explanation of the methods is given here; more detailed information are available from your Milestone local supplier up on request.

3.1 US EPA METHOD 3015

- Sample amount 45mL.
- Reagents 2,5mL HNO_3 and 2,5mL HCl when analyzing the solution with FLAA or ICP, 5mL HNO_3 only when analyzing the solution with FLAA, GFAA, ICP or ICP-MS.
- Microwave digestion time 20 minutes.
- Temperature to reach 160°C within 10 minutes and to remain between 165-170°C for other 10 minutes.

3.2 US EPA METHOD 3051

- Sample amount 0,5g.
- Reagent 10mL HNO_3.
- Acid leaching method.
- Microwave digestion time 10 minutes.
- Temperature to reach 170°C within 5,5 minutes and to remain between 170-180°C for the balance of 10 minutes.

3.3 US EPA METHOD 3052

- Sample amount up to 1,0g.
- Basic reagents 9mL HNO_3 and 3mL HF.
- Alternative combinations include HCl (for Erg, BA, SB, Fe and Al determination), H_2O_2 (for organic samples) and H_2O (to slow down exothermal reaction).
- Goal is total sample decomposition.
- Microwave digestion time 15 minutes.
- Temperature to reach 180°C within 5 minutes and to remain between 175-185°C for 10 minutes.

4.0 MICROWAVE ACID DIGESTION OF INORGANIC SAMPLES

Inorganic samples group includes metals, alloys, oxides, ores, rocks, slags, ceramics etc.

4.1 CHEMISTRY

Sample preparation procedure are strongly dependent on the chemical nature of the material to be digested abd on the elements to be determined.
The microwave acid(s) choice is often the same as used with “conventional” methods.

4.2 MICROWAVE PROGRAM

Most inorganic samples show non-exothermal reactions and do not produce large amount of gases.
The sample amount does not affect the ratio temperature/pressure as much as with organic samples.
The digestion efficiency strongly depends on the digestion temperature; therefore the target is to bring the solution to the highest temperature in the shortest time (first step) and to hold this temperature until the digestion is complete (second step).
For difficult samples, requiring temperatures higher than 200°C, a high pressure rotor MDR-1000/6S should be used, while for easier samples, requiring temperature below 200°C, a medium pressure rotor MDR-300/10S is enough.
As a matter of fact, when samples such as sand (1g) are to be digested with 4mL HF and 1mL HNO_3, a suitable program would be:

Step	Time (minutes)	Power (Watt)
1	10	650
2	10	350

The above program refers to the simultaneous digestion of 6 samples.
A library of nearly 400 sample preparation methods is available from your local Milestone distributor.